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Micron 30 (1999) 197–204

**micron**

www.elsevier.com/locate/micron

# A review of focused ion beam milling techniques for TEM specimen preparation

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Received 4 October 1998; accepted 4 February 1999

## Abstract

The use of focused ion beam (FIB) milling for the preparation of transmission electron microscopy (TEM) specimens is described. The operation of the FIB instrument is discussed and the conventional and lift-out techniques for TEM specimen preparation and the advantages and disadvantages of each technique are detailed. The FIB instrument may be used for rapid site-specific preparation of both cross-section and plan view TEM specimens. © 1999 Elsevier Science Ltd. All rights reserved.

**Keywords:** Focused ion beam; Transmission electron microscopy (TEM); Scanning electron microscopy (SEM); Secondary ion mass spectrometry (SIMS)

## 1. Introduction

In the past few years, we have observed an increase in the use of the focused ion beam (FIB) tool for the preparation of transmission electron microscopy (TEM) specimens as witnessed by an increase of publications in this area. An FIB instrument may be simply referred to as a “fib” in common parlance. While this technique is still in its infancy, many investigators have utilized this method for preparing electron microscopy specimens from a wide range of materials including semiconductors, metals, ceramics, polymers, biological materials, and tissues.

## 2. The focused ion beam instrument

A FIB instrument looks and operates much like a scanning electron microscope (SEM). Both instruments rely on a focused beam to create a specimen image; an ion beam for the FIB and an electron beam for the SEM. For both instruments, the intensity of the secondary electrons produced at each raster position of the beam is displayed to create an image of the sample. In the FIB, secondary ions may also be detected and used to construct an image of the sample. Images having magnifications up to  $\sim 100\,000$  times are available using a FIB with a very good depth of field. The

operation of a FIB begins with a liquid metal ion source (LMIS). A reservoir of gallium (Ga) is positioned in contact with a sharp Tungsten (W) needle. The Ga wets the needle and flows to the W tip. A high extraction field ( $>10^8$  V/cm) is used to pull the liquid Ga into a sharp cone whose radius may be 5–10 nm. Ions are emitted as a result of field ionization and post-ionization and then accelerated down the FIB column. The use of Ga is advantageous for two reasons: (i) Ga has a low melting point and, therefore, exists in the liquid state near room temperature, and (ii) Ga can be focused to a very fine probe size ( $<10$  nm in diameter). FIBs typically operate with an accelerating voltage between 5 and 50 keV. By controlling the strength of the electrostatic lenses and adjusting the effective aperture sizes, the probe current density (and therefore beam diameter) may be altered from tens of pA to several nA corresponding to a beam diameter of  $\sim 5$  nm to  $\sim 0.5$   $\mu$ m). A schematic diagram of the LMIS and FIB column is illustrated in Fig. 1.

An understanding of the sputtering process is important for a knowledgeable operation of the FIB. When a  $\text{Ga}^+$  ion is accelerated toward the target sample, it enters the sample and creates a cascade of events which results in the ejection of a sputtered particle (which may be an ion or a neutral atom). This sputtering mechanism thus also results in  $\text{Ga}^+$  implantation into the sample. The primary ion penetration depth is  $\sim 20$  nm for 25 keV  $\text{Ga}^+$ . The use of enhanced etching may increase the sputtering rate. Halogen gases such as  $\text{Cl}_2$ ,  $\text{I}_2$ , or  $\text{XeF}_2$  can be directed to the area of interest.

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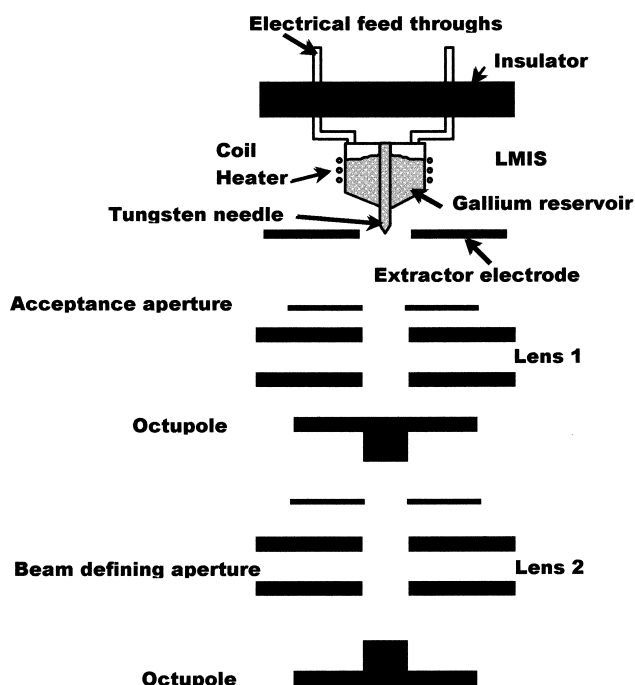


Fig. 1. A schematic diagram of the LMIS and lens system of an FIB (courtesy of B.I. Prenitzer).

These gases form a volatile compound with the sputtered material, thereby increasing the sputtering rate.

Another feature that is used extensively in the FIB is ion beam assisted chemical vapor deposition. The deposition of metal is used extensively in silicon semiconductor device modification and is used in both SEM and TEM specimen preparation techniques to protect the top surface of interest from spurious sputtering. A needle is brought to within  $\sim 100\text{--}200\text{ }\mu\text{m}$  of the target surface. A suitable gas (e.g.,  $\text{W}(\text{CO})_6$ ) is injected from the needle and adsorbs onto the target surface. The  $\text{Ga}^+$  beam is rastered over the desired specimen region. The ion beam decomposes the gas, which leaves a deposited layer of metal (e.g., W), while the by-product (e.g., CO) is removed through the vacuum system. Other conductors (e.g., Pt, Al, Cu, and C) and insulators (e.g.,  $\text{SiO}_2$ ) have also been used as deposition materials.

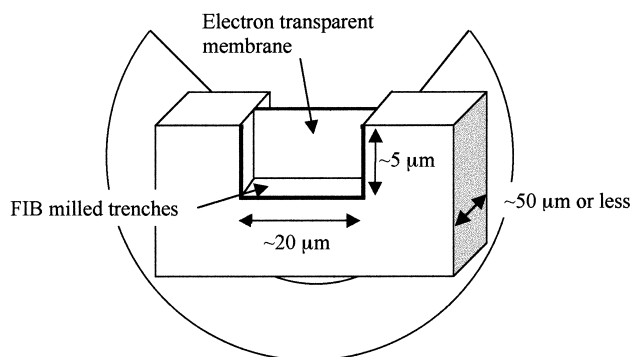


Fig. 2. A schematic diagram of a conventionally prepared FIB TEM specimen (not drawn to scale).

Sample charging of insulating materials may be reduced by coating the sample surface with carbon prior to insertion into the FIB. In some instruments, an electron flood gun is available for charge neutralization. In addition, a mass analyzer, usually a quadrupole, can be attached to the FIB for secondary ion mass spectrometry (SIMS) analysis of the secondary ions. Dual beam instruments, having both an ion column, and an electron column, are also commercially available.

The FIB can provide grain size measurements. The contrast mechanism in the FIB image is a result of ion channeling through different crystallographic directions in the different grains which are exposed to the ion beam at different angles of incidence. Therefore, grain size measurements may be routinely determined by tilting to several angles of incidence and overlaying the respective images to determine the grain boundary locations accurately.

The microelectronics industry continues to push the advancements in FIB instrumentation. However, the FIB is being realized as a general characterization and specimen preparation tool for both the physical and biological sciences. The small beam size and imaging capabilities of the FIB make this instrument ideal for preparing site-specific SEM or TEM specimens in either cross-section or plan view. The beam can be very accurately positioned on the sample at high current density to produce large (i.e.,  $20\text{ }\mu\text{m} \times 5\text{ }\mu\text{m}$ ) uniformly thin TEM specimens (e.g.,  $<100\text{ nm}$ ) that may be prepared in just a couple of hours. In the following, we shall describe two techniques for preparing TEM specimens. The FIB is particularly suited for cross-section TEM specimen preparation as FIB milling occurs sequentially through each layer. Therefore, the problem of preferential milling between different materials is nearly negligible. However, the FIB is equally useful in preparing plan view specimens of individual thin film layers. We shall refer to the first method described as the "conventional" FIB method and the second method described shall be referred to as the "lift-out" technique.

### 3. Conventional FIB TEM specimen preparation

#### 3.1. Cross-section method

Most of the literature listed in the reference section refers to the conventional FIB method of TEM specimen preparation. A detailed description of this method is given by Stevie et al. (1995). Initial specimen preparation must be performed before the sample is placed into the FIB using the conventional TEM specimen preparation method. An area of interest is located and cut to  $< 3\text{ mm}$  in length. The sample is then mechanically polished as thin as possible (e.g.,  $< 50\text{ }\mu\text{m}$ ) to reduce the FIB time. It should be noted that tripod polishing is ideal for this application (Anderson and Klepeis, 1997). A microcleaver may also be used to

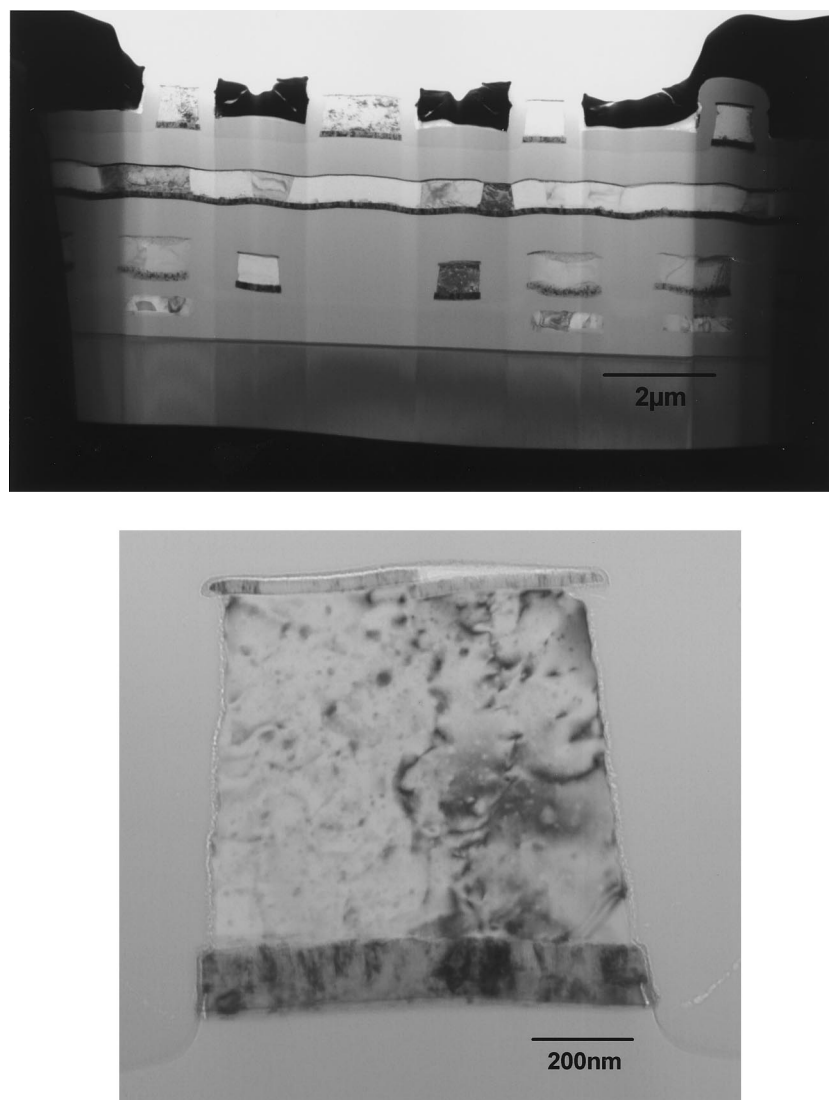


Fig. 3. Low magnification and high magnification cross-section TEM images of a semiconductor specimen prepared by the conventional FIB method. (courtesy of R.B. Irwin and J.L. Drown-MacDonald).

prepare thin sections of brittle materials for FIB thinning. Samples may also be cut to size using a wire saw. The sample is mounted on a slotted (e.g.,  $1\text{ mm} \times 2\text{ mm}$ ) TEM Cu grid that has been partially cut away (see Fig. 2). The sample is then positioned into the FIB so that sputtering may begin.

A metal line is usually deposited on the area of interest to prevent damage and spurious sputtering of the top portion of the specimen and to also delineate the location of the area of interest. Typical dimensions of the metal line are  $\sim 1\text{ }\mu\text{m}$  wide  $\times 2\text{ }\mu\text{m}$  high  $\times 30\text{ }\mu\text{m}$  long. Large trenches are sputtered on either side of the area of interest using a high  $\text{Ga}^+$  beam current. To reduce stress in the thin membrane, a spring may be micromachined into the sample at this time (Walker, 1998). The beam current is reduced and milling is performed on alternate sides of the specimen to reduce redeposition of sputtered material onto the surface of the specimen. Milling is continued until the membrane is thinned to

$\sim 100\text{ nm}$  or less (the final thickness of the specimen will depend on the information sought and the density of the material(s)). A finished electron transparent portion of the sample is usually  $\sim 5\text{ }\mu\text{m} \times 20\text{ }\mu\text{m}$ . An example of a TEM specimen prepared by the conventional FIB technique is shown in Fig. 3. The dark contrast on the left, bottom, and right portion of Fig. 3(a) are the trench walls. The “curtains” or vertical stripes which appear as light and dark contrast in the micrograph of Fig. 3(a) form due to changes in sputtering rates due to differences in specimen topography. Fig. 3(b) is an enlarged area of Fig. 3(a) which shows Ti/TiN layers on both the top and bottom of an Al line surrounded by  $\text{SiO}_2$ . A TEM specimen of a Si-based integrated circuit may be prepared in less than 1 hour. Multiple TEM specimens may be milled into the same piece of “bulk” material. This is very useful for analysis of serial sectioning or multiple plan view analyses (Saka et al., 1996; Anderson and Klepeis, 1997).

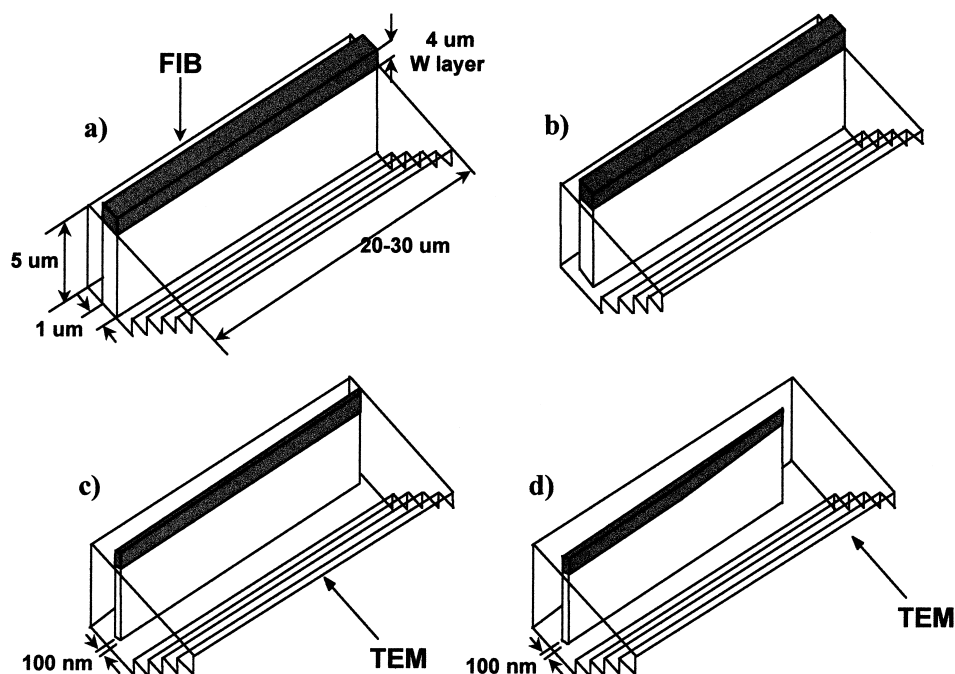


Fig. 4. A schematic diagram of the FIB lift-out technique (not drawn to scale). See text for description.

### 3.2. Plan view method

The preparation of plan view specimens is similar to that for cross-section specimens (Anderson and Klepeis, 1997). The plan view method essentially requires that the bulk sample be oriented so that planar interfaces are positioned parallel to the ion beam. For the preparation of TEM plan view specimens, the top edge of the specimen that contains the region of interest is polished (or cleaved) and the bulk sample is cut and/or polished to size using the same techniques mentioned previously. The mounted sample is then positioned into the FIB so that the plane of interest is parallel to the ion beam. An electron transparent membrane is then FIB milled in a similar manner for the preparation of cross-section specimens.

## 4. The FIB “lift-out” technique

The lift-out technique requires little or no initial specimen preparation (Overwijk et al., 1993; Leslie et al., 1995; Herlinger et al., 1996; Stevie et al., 1998; Sheng et al., 1997; Giannuzzi et al., 1997a,b). The only requirement for the lift-out technique is that the bulk sample must fit inside the FIB specimen chamber. Insulating materials are sometimes pre-coated with carbon or chromium to prevent charging. In the lift-out technique, the electron transparent thin membrane is actually removed from the bulk specimen and analyzed directly by TEM. A metal line is usually deposited over the area of interest as in the conventional FIB method. A large stair-step FIB trench is cut on one side of the area of interest and a rectangular FIB trench is cut on the other side

of the area of interest (Fig. 4(a)). Prior to final thinning, the sample is tilted to  $>45^\circ$  and then the bottom, left side, and a portion of the right side of the specimen is cut free (Fig. 4(b)). Then the sample is tilted back to its starting position and the specimen is thinned to electron transparency as mentioned earlier (Fig. 4(c)). If the specimen is to be used for high resolution electron microscopy (HREM), a final FIB cut is performed  $\sim 1\text{--}2^\circ$  with respect to the plane of the specimen surface. In this manner, the thinnest portion of the specimen lies in the area of interest required for HREM analysis as illustrated in Fig. 4(d) (Giannuzzi et al., in press). The remaining right side of the specimen is milled free leaving the electron transparent membrane lying in the cut trenches. Plan view specimens may also be prepared with the lift-out technique (Stevie et al., 1998).

The bulk sample is removed from the FIB vacuum chamber and is then viewed using a light optical microscope that has an objective lens with a large working distance. A solid glass rod pulled to a sharp tip ( $\sim 20\text{--}30\text{ }\mu\text{m}$ ) is inserted into the arm of a hydraulic micromanipulator. Using the micromanipulator, the electron transparent membrane is “lifted out” of the bulk sample and is then positioned onto a carbon or formvar coated Cu TEM mesh grid. Electrostatic forces allow the membrane to be lifted out by means of the glass rod. The specimen is ready for TEM analysis. Lift-Out TEM specimens may be prepared within 3 h. FIB instruments that employ large beam currents (i.e., tens of nA) allow for faster milling of bulk trench cuts and have reduced the overall milling of a lift-out (or conventional) specimen to  $<1\text{ h}$ . In our lab, we have maintained a lift-out success rate that exceeds 90% for hundreds of samples.

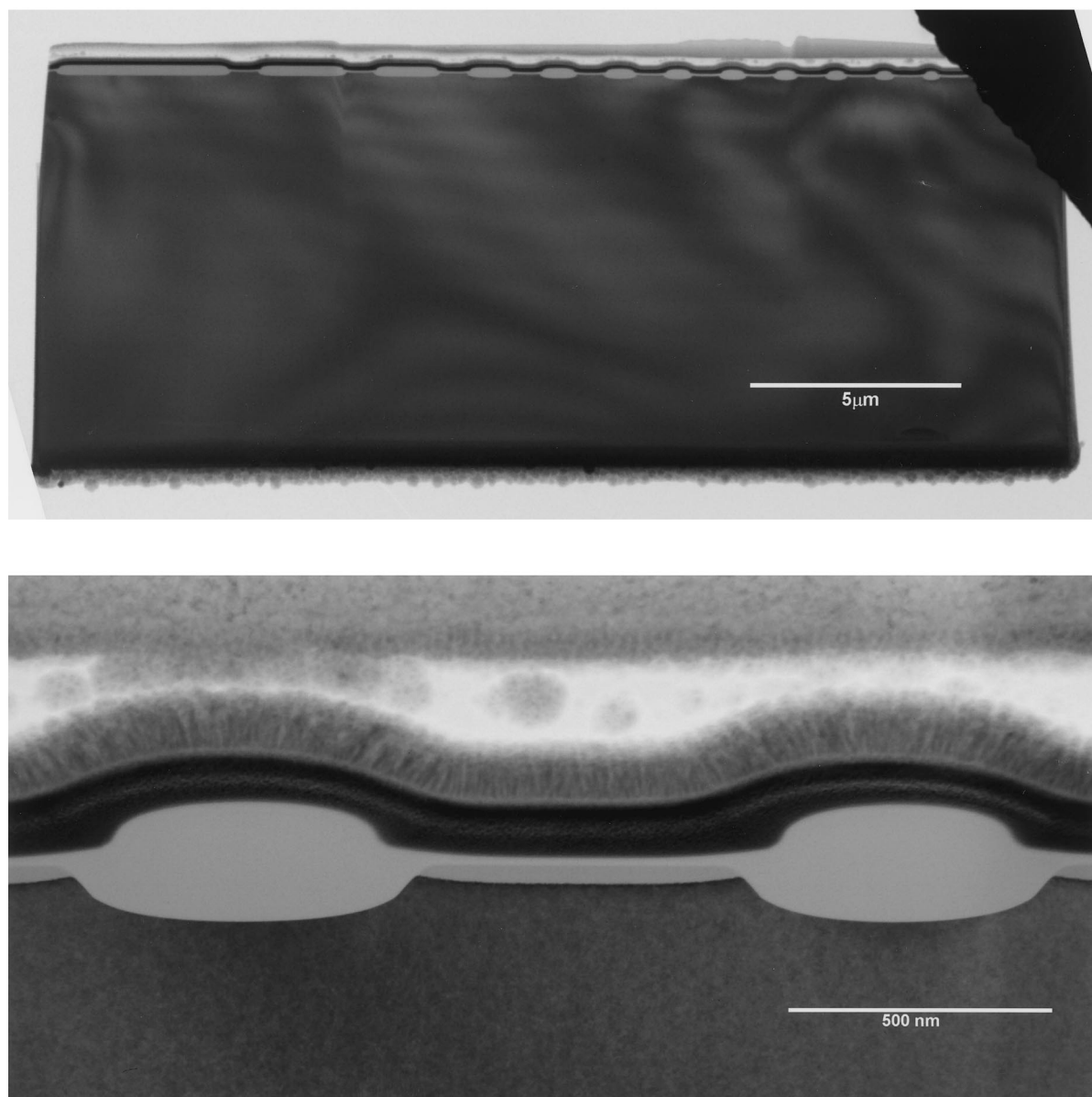


Fig. 5. Low magnification and high magnification cross-section TEM images of a semiconductor specimen prepared by the FIB lift-out technique (courtesy of R.B. Irwin, J.L. Drown-MacDonald).

An example of the lift-out technique used to obtain a site specific cross-section of an integrated circuit is shown in Fig. 5 which includes (a) a low magnification TEM image of a lift-out specimen; and (b) a higher magnification TEM image of the same lift-out specimen.

### 5. FIB preparation tricks of the trade

The preparation of a TEM specimen from a Si-based integrated circuit has become routine. The overall quality of the focused ion milling will be material dependent. The electron transparent membrane walls are not perfectly parallel to each other. A cross-section of any FIB prepared TEM

specimen shows broadening at the base and the angle of this broadening varies with the atomic number. This sputtering phenomenon is known as the “classic V-shape” and its shape is a function of the competition between sputtering and redeposition for a given material. In order to compensate for the V-shape that develops during FIB milling, it may be necessary to tilt each side of the specimen into the beam prior to the final polishing steps. For example, a Zn specimen was tilted  $\pm 14^\circ$  into the beam in order to prepare an electron transparent TEM specimen with near parallel side walls (Prenitzer et al., 1998a–c). We are attempting to model FIB milling characteristics as a function of atomic number so that specific FIB parameters may be determined a priori for any material system (Prenitzer et al., 1998a–c).

## 6. Specimen preparation FIB induced damage

There are well known  $\text{Ga}^+$  beam induced damage artifacts that exist in FIB TEM specimens. Ga ions are implanted and mix into the specimen as a result of the sputtering mechanism. Therefore, it is possible that Ga may influence local compositions within the specimen, although the extent of this effect is not known. Hence, care should be taken when performing analytical microscopy. The Ga implantation also amorphizes the outer layer of the specimen. There is still some uncertainty in the literature as to exactly how much amorphous damage is produced during FIB sputtering (Albarede and Lezec, 1998; Susnitzky and Johnson, 1998; Kamino et al., 1998; Prenitzer et al., 1998a–c). Experimental observations are consistent to within one order of magnitude with Monte Carlo simulations, suggesting that a 25 keV, 1000 pA  $\text{Ga}^+$  beam may produce  $\sim 20$  nm of amorphous damage on each side of the FIB specimen (Prenitzer et al., in press). This large beam diameter would be used in a bulk FIB cut and, therefore, it is reasonable to assume that the final polish cuts would produce less amorphous damage. Some results show that amorphous damage may appear in  $> 100$  nm of the specimen surface (Albarede and Lezec, 1998). This implies that the entire specimen would become amorphous during FIB sputtering; however, it has been shown that high resolution lattice images of crystalline materials can be achieved from FIB prepared specimens quite readily using both the conventional and lift-out techniques (Tarutani et al., 1992; Tsuji et al., 1996; Bender et al., 1997; Tsujimoto et al., 1997a,b; Susnitzky and Johnson, 1998; Giannuzzi et al., in press; Kamino et al., 1998; Phaneuf et al., 1998). Experience in producing quality specimens with the FIB is necessary as with any TEM specimen preparation technique.

## 7. A comparison of the FIB methods

The primary advantage that is common to both methods is that the FIB tool allows for rapid production of site specific (to within  $\sim 200$  nm) TEM specimens. The disadvantages common to both techniques are the FIB induced artifacts described before.

The conventional FIB method requires significant (e.g., destructive) initial specimen preparation before insertion into the FIB, while the lift-out technique requires little or no initial specimen preparation. Thus, the lift-out specimen may be used for serial sectioning of a bulk material. In addition, a bulk sample may be processed or altered in many ways and a lift-out TEM specimen may be extracted from the same bulk sample after each subsequent processing step.

Conventional FIB specimens suffer from reduced TEM tilt capability due to the presence of the bulk specimen side-walls. The tilting of a lift-out specimen is only limited by the capabilities of the TEM and not the specimen itself. In

addition, the side walls in conventional FIB specimens produce a large amount of spurious X-ray fluorescence during energy dispersive spectroscopy analysis [Saito et al., 1998; Longo et al., 1998]. Increasing the trench size length and shape in a conventional FIB specimen significantly reduces X-ray fluorescence during EDS analysis. This extra FIB milling obviously increases the specimen preparation time.

Experience in our laboratory has shown that care must be taken when manipulating the electron beam while imaging a lift-out specimen in the TEM. It is our experience that an increase in electron dose exposed directly to a lift-out specimen may cause unwanted specimen damage and/or phase transformations due to the lack of a heat sink. This merely requires that the TEM operator move the specimen out of the field of view of the electron beam when increasing the spot size and realigning the beam.

If the prepared specimen is too thick, a conventionally prepared specimen may be placed back into the FIB for further thinning, while currently the lift-out technique does not permit further thinning. Hence, another FIB specimen must be milled which may not be viable for “one-of-a-kind” type samples. As mentioned earlier, both techniques are suitable for high resolution TEM analysis.

An interesting advantage for FIB TEM specimens is the ability to map out compositional dopant variations at the 10 ppm level. This has been observed for InP-based and similar materials (e.g., InGaAs, InGaAsP) prepared only by the FIB specimen preparation techniques (Hull et al., 1995, 1998; Kimura and Shimuzi, 1997). It is believed that the absorption contrast observed centers upon clustering of P point defects in differently doped layers associated with isoelectronic  $\text{Ga}^+$  centers introduced by the FIB.

## 8. Advances in FIB instrumentation

Manufacturers that produce FIB instruments which we are aware of include (in alphabetical order): FAI, FEI/Philips, Hitachi, JEOL, Metron/Seiko, Micrion, NANO-FAB, and Schlumberger. Equipment manufacturers are exploring the introduction of liquid metal ion sources other than Ga. Special TEM/FIB specimen holders and instruments are available so that conventional specimens can be produced and alternated between instruments without additional handling concerns (see e.g., Gatan Inc., Hitachi). Liquid metal ion sources have also been incorporated into a TEM for direct imaging and specimen preparation (Tarutani et al., 1993). A light optical microscope is now available for the lift-out technique that has only one large working distance objective lens that covers the full range of available magnification (see e.g., Micro-Optics of Florida).

As ions are used as the source in the FIB, sputtering of the sample will occur whenever the beam is “on.” Therefore, damage of the thin electron transparent membrane is possible just by observing it in the FIB. Hence, dual beam

instruments are now available that have both an electron gun and an ion gun (albeit at a greater expense). In this manner, observation of the specimen can be achieved with the electron source, and sputtering will occur only when the ion gun is on. Automation of the bulk trench cuts and most of the polish cuts are now possible with new FIB systems. Multiple specimen holders for the FIB are also available so that the bulk cuts of several samples may be achieved quickly and without operator intervention. It is conceivable that in the future routine TEM specimen preparation times may be reduced to < 30 min!

## 9. Conclusions and final comments

An FIB tool may cost between 30 and 80% of the total cost of a TEM. This is a lot of money to spend for a specimen preparation tool, considering that the TEMs we use to analyze these specimens cost the same order of magnitude. However, materials systems are becoming more complex, and the increasingly smaller critical dimensions of features necessitate the use of the FIB. The use of the FIB is slowly expanding outside the microelectronics industry and into the “general” industrial population and university laboratories. There is much work to be done and many more applications to discover with the FIB. The FIB TEM specimen preparation methods are in their infancy. We are learning that the TEM analyses of some materials that seemed difficult and/or impossible in the past are presently being realized. However, as with any TEM specimen preparation technique, it is important to understand the limitations and possible artifacts within the specimen, and to interpret the TEM results accordingly.

## Acknowledgements

The support of the following agencies is greatly acknowledged: NDSEG Fellowship P-34862-RT-NDF, NSF Award 9703281, the I4 High Tech Corridor Council, Enterprise Florida, Cirent Semiconductor. We would also like to thank the support of Jeff Bindell, who has been a proponent of the UCF/Cirent partnership. We have been fortunate to collaborate with the following Cirent staff and UCF students: Steve Brown, Jennifer Drown-MacDonald, Rich Irwin, Brian Kempshall, Brenda Prenitzer, Teri Shofner and Carrie Urbanik.

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