Transmission Electron Microscope Specimen Preparation of Zn Powders Using the Focused Ion Beam Lift-Out Technique

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Particles of Zn powder have been studied to show that high-quality scanning electron microscope (SEM) and transmission electron microscope (TEM) specimens can be rapidly produced from a site-specific region on a chosen particle by the focused ion beam (FIB) lift-out technique. A TEM specimen approximately 20-μm long by 5-μm wide was milled to electron transparency, extracted from the bulk particle, and micromanipulated onto a carbon coated copper mesh TEM grid. Using the FIB lift-out method, we were able to prepare a site-specific TEM specimen from a difficult material in under 3 hours. The TEM analysis of the lift-out specimen revealed a large amount of thin area free from characteristic signs of damage that may be observed as a result of conventional argon ion milling. The overall microstructure of the specimen prepared by the FIB lift-out method was consistent with samples prepared by conventional metallographic methods. A grain size of ~10 to 20 μm was observed in all specimens by both TEM and SEM analysis. Light optical microscopy revealed the presence of internal voids in ~10 to 20 pct of all particles. The SEM analysis showed the voids to extend over ~70 pct of the particle volume in some cases.

I. INTRODUCTION

Powder metallurgy (P/M) is a technique in which fine metal particles, typically less than 100 μm in dimension, are used as feed stock in the production of high-quality, low cost, or otherwise unique components. Advanced forming techniques, in conjunction with small particle size, have made nonequilibrium microstructures such as glassy, nano- and microcrystalline metals attainable. Premixed and prealloyed powders can be used to exert control over a product's final compositional and microstructural characteristics, thus avoiding problems such as coarse, dendritic second-phase segregation. The P/M technique has proven to be effective in the production of specialty items for nuclear, aerospace, electrical, and magnetic applications.[1–4]

Mechanical, electrical, and magnetic properties of engineering components can all be traced back to the microstructural characteristics of the component. Although compaction and sintering mechanisms are critical to the manufacture of fully dense, near-net shape parts,[5,6,7] the performance of the final product may be limited by the material properties of the individual particles. A part fabricated by advanced P/M methods will possess a level of microstructural integrity comparable to the powder from which it was formed. Thus, it is important to examine the microstructure of the individual particles for point, line, and planar defects and features such as porosity, inclusions, cracking, and segregation. Light optical microscopy (LOM) and scanning electron microscopy (SEM) are useful for observing macroscopic defects. In some cases, SEM can also be used to determine grain boundary structure and orientation relationships; however, transmission electron microscopy (TEM) is the preferred technique for performing detailed microstructural evaluation and defect analysis.

A TEM specimen should be representative of and unchanged from the bulk microstructure. If the microstructure is modified during specimen preparation, then those changes should be well characterized. A typical TEM specimen is 3 mm in diameter having electron transparent regions on the order of a few μm wide by ~100 nm in thickness. The dimensional requirements alone are enough to ensure that the preparation of TEM specimens by conventional methods will be nontrivial. The TEM specimen preparation becomes more rigorous when the sample geometry or composition is complex. For example, fibers, powders, composites, and interfacial regions require special consideration.[8,9] Individual fibers and powders are too small to be prepared as a self-supporting specimen; therefore, particulate materials must be embedded in some type of binder prior to conventional thinning techniques. As a result, TEM specimen preparation is further complicated by the fact that the bulk particle/binder sample must now be treated as a composite material where the two phases may have very different material properties.

The Zn powder particles chosen as the subject of this investigation are doubly formidable. In addition to the small particle geometry, the chemical nature of Zn makes it a difficult material to prepare for TEM. The Zn test material was specifically selected for the lift-out technique because of its notoriously low damage threshold. The low Zn melting temperature of 693 K is an indicator of the relatively weak interatomic bonding forces that make it especially susceptible to recrystallization and internal defect generation.

The successful preparation of a TEM specimen is often

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Manuscript submitted January 15, 1998.

Fig. 1—An SEM image of Zn powder particles mounted in M-bond epoxy.

the limiting factor in performing TEM analysis. Traditionally, it has been the case that, when developing a strategy for the preparation of a specific TEM specimen, each material system had to be treated uniquely. Nevertheless, the analytical potential of the TEM continues to drive the development of new and innovative specimen preparation methods. As materials become more complex, specimen preparation becomes increasingly difficult. Complex compositions, geometries, and the need to sample specific regions present new frontiers. An ideal preparation method should be capable of rapidly producing high-precision, site-specific specimens from virtually any material system with a minimum amount of parametric modification.

Focused ion beam (FIB) milling is a technique that has many applications.[10–19] A finely focused, energetic beam of ions has proven to be very useful as a sputtering/deposition tool in micromachining and microfabrication applications. In particular, the site-specific milling/deposition capability of the FIB has found great utility in the area of device modification within the microelectronics industry.[14,16,20–26] The secondary electron imaging capability of the FIB can be used to pinpoint a specific region of interest for milling or to perform an *in situ* microstructural evaluation. A secondary ion mass spectrometer (SIMS) can also be added to perform elemental analysis and obtain ion images of small sample areas.[27,28] The analytical capacity of the FIB has been extended in some of the newer instruments by the integration of a fully functional SEM column. Because the electron and ion beams converge at the same point on the sample, the dual beam instruments allow for nondestructive, enhanced process monitoring by providing high-resolution, real-time SEM images. In addition to its potential as a stand alone analytical tool, the milling capability of the FIB has been extended to the preparation of samples for subsequent analysis by dedicated SEM, TEM, and SIMS instrumentation. Special attention has been given to the preparation of cross section and other challenging TEM samples by FIB milling.[10,12,14,16–19]

There have been a number of iterations and variations of the FIB TEM specimen preparation technique. Yasuyuki *et al.* describe a method for the preparation of micrometer-sized powder particles by FIB milling.[17] Their method involves mounting particles onto the flat, thin edge of a semicircular 3-mm tantalum disc and then thinning the entire diameter of the particle to electron transparency. They report an amorphous region extending through the top 5 nm of the particles, probably the direct result of Ga^+ milling damage.[29] In addition to surface damage, this method involves removal of large amounts of material, which translates into unnecessarily long milling times.

A group from Philips Research Laboratories first described a method in which a thin electron transparent membrane was milled in the FIB, extracted from the bulk material, and mounted on a standard TEM specimen holder.[12] Work recently performed in our laboratory has expanded on the work done at Philips. Giannuzzi *et al.* describe in detail the lift-out method for site-specific cross-sectional TEM specimen preparation.[19] The work done by our group has served to extend the FIB lift-out technique to a wide range of material systems and geometries.[18]

II. SPECIMEN PREPARATION OF ZN POWDERS

A. *Metallography*

All SEM analysis in this work was performed on a Hitachi H-4100 operating in secondary electron imaging mode at 6 kV and a working distance of 19 mm. A Nikon Optiphot operating in reflective mode was used for the LOMs.

Commercially available Zn powder, fabricated by the air atomization of refined Zn, was acquired for this study courtesy of Keystone Powdered Metal Company. The powder particles were dispersed in either a quick drying epoxy or M-bond[30] in an approximate 1:1 ratio by volume. A droplet of the suspension was deposited onto either a small piece of Cr-plated Si wafer or onto the surface of an Al SEM stud. A thin layer of the suspension was deposited to ensure that some of the particles would protrude from the epoxy surface. All samples were then allowed to cure at room temperature. No significant differences in performance or analysis were noted between the two substrate materials; however, all of the samples reported on in this work were fixed with M-bond to a Cr-plated Si substrate. Figure 1 is an SEM image of the particles mounted, as described earlier. The particles shown are typical of the dimensions that were selected for FIB specimen preparation. The particles are roughly ligamental in shape, 50- to 100 μm along the major axis with an aspect ratio of slightly greater than 3:1. This particle shape is expected and is typical of powders prepared by air atomization.[31]

Additional powder samples, mounted (as described earlier), were prepared using conventional metallographic techniques. These samples were intended to serve as standards from which to assess potential microstructural damage that may be induced by the FIB milling process. Samples were mechanically polished using a tripod polisher on water-lubricated diamond lapping films with successively finer grit sizes of 6, 1, and 0.5 μm. The final polishing step was performed on a felt polishing cloth using a suspension of 0.05 μm Al_2O_3 particles in distilled water. Figure 2 is an LOM image showing a cross-sectional view of the Zn particles, as prepared by conventional metallographic methods prior to etching. Note the presence of internal porosity and the large distribution of particle sizes and shapes within the LOM micrograph in Figure 2. This

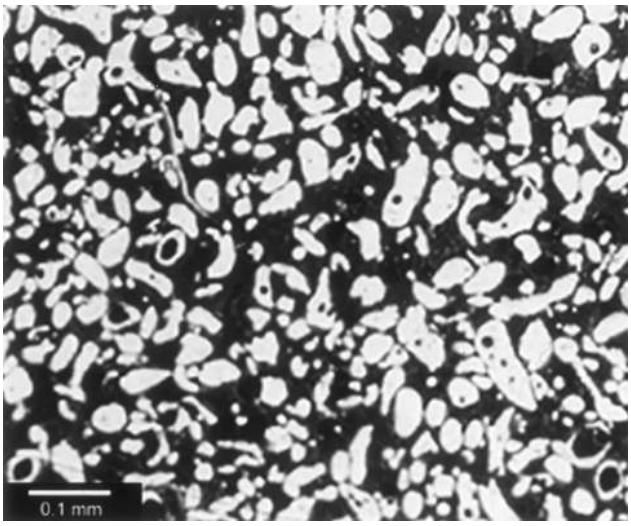
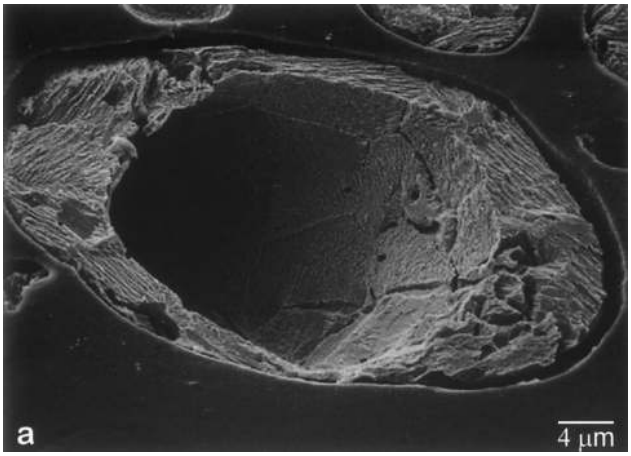


Fig. 2—An LOM image of polished Zn powder particles.



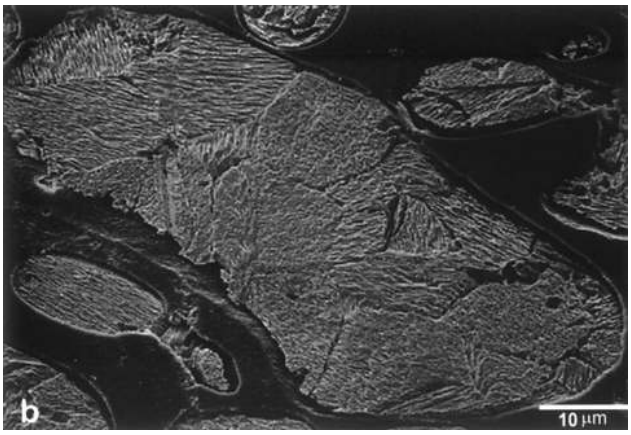


Fig. 3—(*a*) and (*b*) SEM images of polished and etched Zn powder particles. Note the large pore within the Zn powder in (a).

type of internal pore structure is also consistent with the air atomization process because gas pockets are frequently trapped within solidifying particles.[31] Figures 3(a) and (b) are SEM images of metallographically prepared powders that were subsequently etched in concentrated HCl. Figure 3(a) shows the inside of an exaggerated pore extending through the majority of the particle. Individual grains ~10 μm in dimension are observed on the inside of the pore that extends over ~70 pct of the particle volume. Figure 3(b) shows a polished and etched Zn particle devoid of pores. Note that the grain morphology is approximately equiaxed, consisting of large grains between 10 and 20 μm in dimension.

B. *The FIB Instrument*

The SEM and TEM specimens were milled with an FEI 611 FIB workstation. In the FIB, Ga is field evaporated from a liquid metal ion source (LMIS),[32] postionized, and accelerated down the column to a kinetic energy between 20 and 30 keV. Two electrostatic lenses focus the energetic ions to a fine probe. A probe diameter of <200 nm is readily attainable in the 611 even at beam currents as high as 2000 pA.

The specimen chamber in the FIB is large enough to accommodate bulk samples up to 152 mm in diameter and 25 mm in height. The range of motion of the high-precision sample stage includes *x*, *y*, and *z* translation, 360-deg rotation about the axis perpendicular to the stage surface, and tilting as great as 60-deg in one direction. The FIB is rastered over the sample surface using two octupole deflectors. Raster fields, ranging from 1 mm to smaller than 1 μm, are stored as algorithms in the instrument's computer memory. Any combination of the predefined raster patterns can be selected, and the sample stage may be positioned over the region of interest with an accuracy of better than 0.1 μm.[16] The high level of control over the raster field in conjunction with the secondary electron imaging capabilities make the Ga LMIS FIB system ideal for site-specific ion milling. It has been shown that material can be sputtered from a region with a lateral resolution better than 0.1 μm.[16] In addition to material removal, the FIB system can achieve similar resolution for metal deposition using ion beam-assisted chemical vapor deposition.[33]

C. *SEM Specimen Preparation*

A single particle was selected for subsequent SEM specimen preparation using the imaging capability of the FIB. Prior to milling an SEM or TEM specimen, a protective W line ~20-μm long by 0.4-μm wide by 4-μm thick is deposited over the region of interest. The material W sputters at a much slower rate than Zn and, therefore, can act as a barrier to protect the region of interest from the deleterious effects of spurious sputtering. A beam current of 500 pA was used to mill a single stair-step trench on the front side of the W line for the SEM specimen. A typical stair-step trench, illustrated by the schematic diagram in Figure 4, is ~24-μm long by 10-μm wide by 7-μm deep at the region of interest directly under the W line. In order to obtain a flat surface, the area to be analyzed using SEM is smoothed by milling in a series of steps at reduced beam currents. The SEM specimen shown in Figure 5 is tilted so that the milled surface makes a ~60-deg angle with the electron beam. At this orientation, the W line is clearly visible across the top surface, and the stair-step trench can be seen in front of the region of interest. The vertical lines on the specimen face are a topographical artifact known as curtains. Curtains are caused by the sputtering action of the Ga^+ ions on a surface that is parallel to the incident beam. This effect can be minimized by additional milling steps

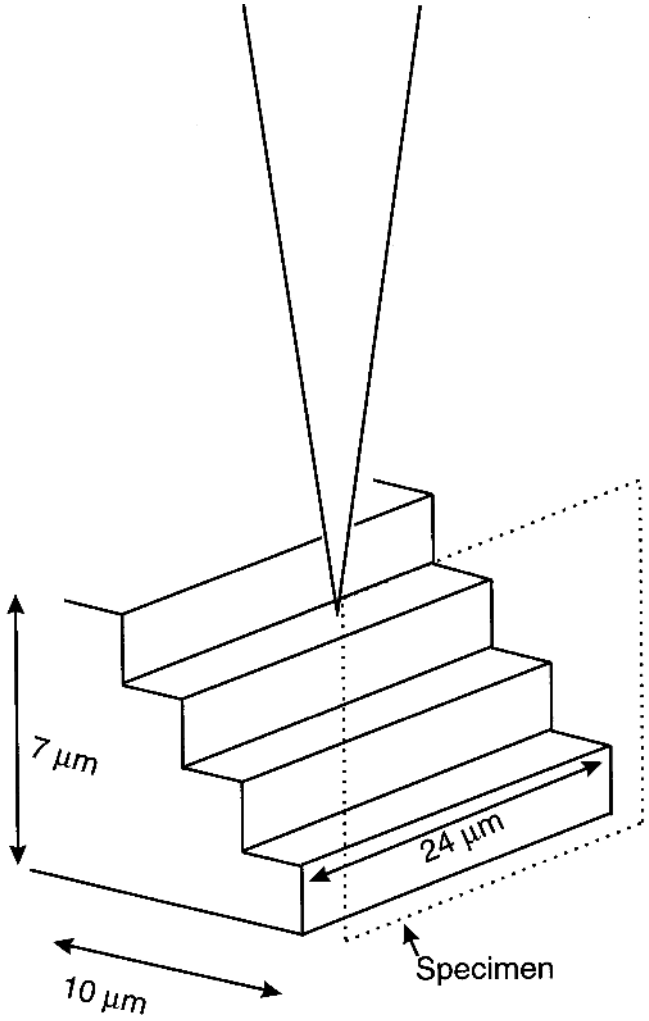


Fig. 4—A schematic diagram of a single stair step trench.

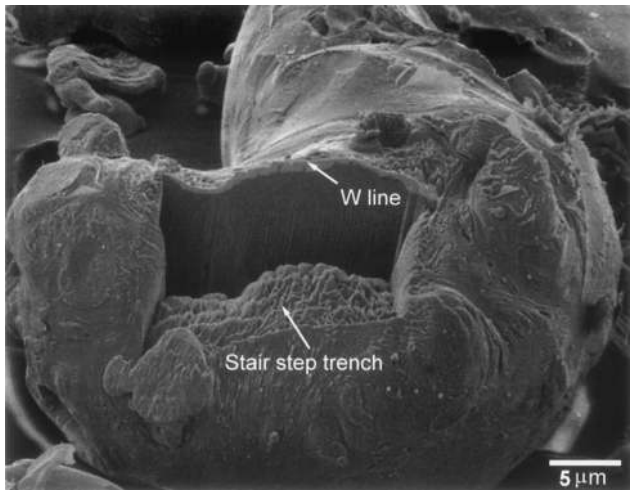


Fig. 5—An SEM image of an FIB SEM cut on a Zn powder particle.

after a slight rotation of the bulk sample about an axis normal to the specimen surface. This specific region on the particle shown in Figure 5 was selected, FIB milled, and ready for insertion into an SEM within 1 hour. The success rate for milling SEM specimens is virtually 100 pct.

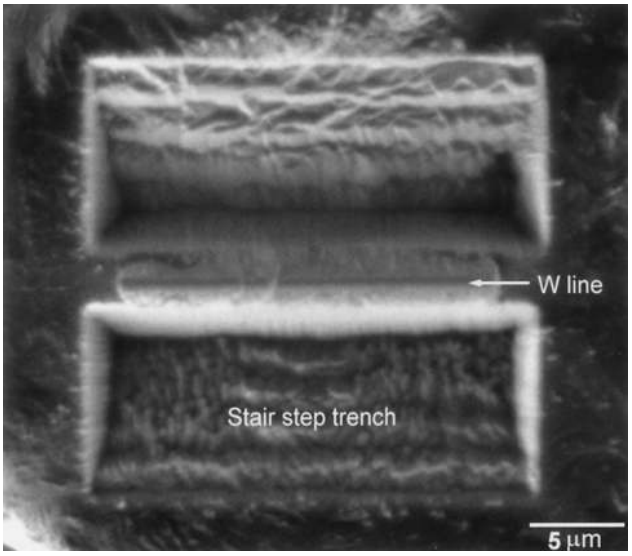


Fig. 6—An FIB image of the double stair step trench produced in a Zn powder particle. Note the sloped sidewalls near the deepest portion of the trench adjacent to the W line.

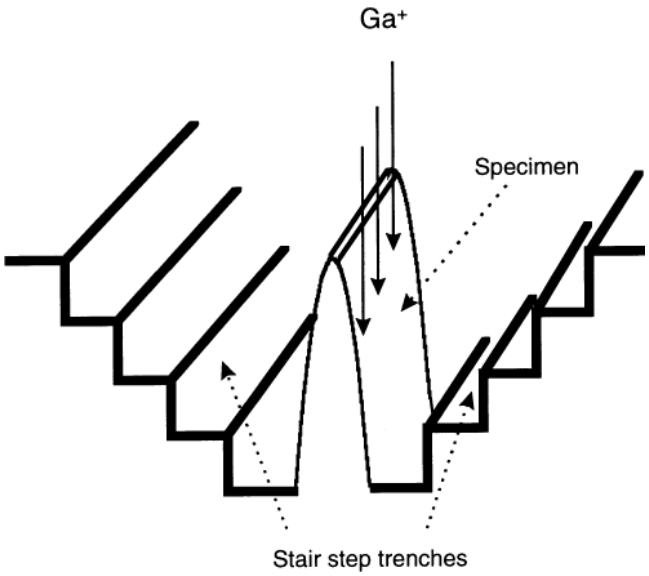


Fig. 7—A schematic diagram of the milled sloped sidewalls known as the ''classic V shape.''

D. *TEM Specimen Preparation*

The sample shown in Figure 5 was specifically prepared for SEM analysis; however, the FIB preparation of an SEM specimen is analogous to the initial steps of the TEM lift-out technique. Prior to milling the TEM specimen, a W line was deposited to mark and protect the particular region of interest. Two stair-step trenches, one on either side of the W line, were then milled at a beam current of 500 pA. The stair-step trenches are milled on both sides of the W coating to facilitate tilting the specimen into the Ga^+ beam during subsequent milling, as described later. Figure 6 is an FIB image of a powder particle observed from the direction parallel to the milling direction after the initial stage of milling at high beam current. When the particle is observed from this vantage point, an exaggerated sloping of the sidewalls on either side of the W line is evident, as illustrated in the

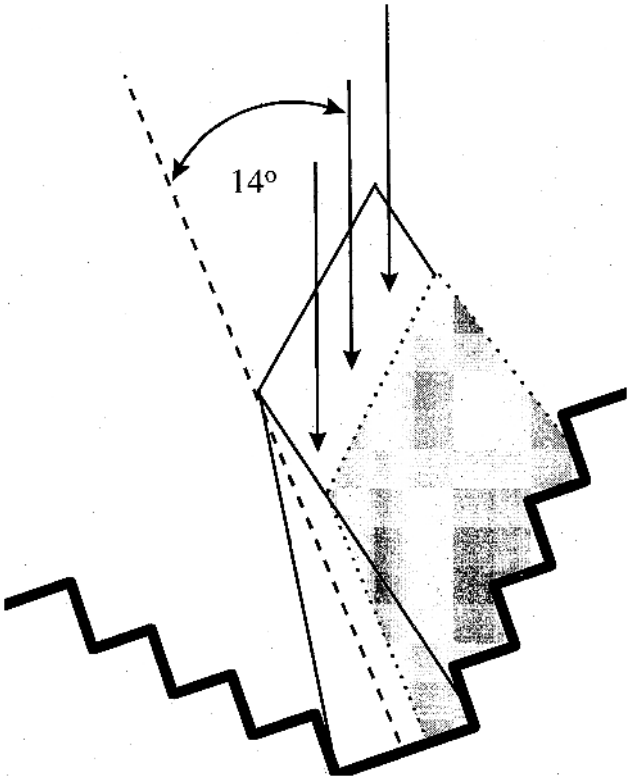


Fig. 8—A schematic diagram showing the 14 deg tilt used to offset the sloped milling of the specimen sidewalls.

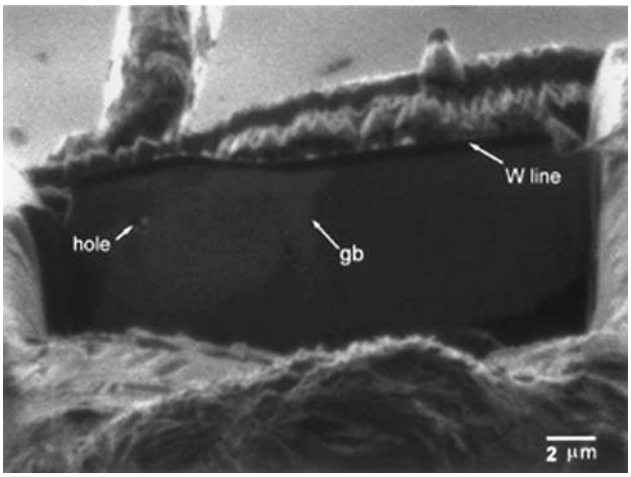


Fig. 9—An FIB image of a TEM specimen tilted at 60 deg prior to completion of milling and lift out.

schematic diagram in Figure 7. This phenomenon has been referred to in the literature as the classic V shape.[13–15,34–36] A hole milled with an FIB tends to be wide at the top surface and tapers down to a point at the bottom of the hole. This gradual sloping has been attributed to the redeposition of sputtered material when milling at high beam currents.[14,34–36] As the hole gets deeper, the effects of redeposition become increasingly severe until the rate of redeposition equals the rate of sputtering, thus limiting the aspect ratio. We have noted that the degree of severity of the V shape shows some material dependency, because it is much more pronounced in metals than in silicon-based materials. Redeposition can be counteracted by the local introduction of a reactive gas species to the milling area.[14,35,36] The gas combines with the sputtered material allowing it to be volatilized and removed by the vacuum system. Although reactive gas-enhanced etching is an attractive solution to the problems resulting from redeposition, the gas has the potential to react with the sample material; therefore, we have opted to offset the initial V-shaped milling using geometrical considerations for subsequent milling operations. After making the initial stair-step trenches, the sample was tilted into the beam, as illustrated in Figure 8.[13,14,15] Tilting the sample changes the angle of incidence so that vertical sidewalls can be milled, and ideally, a uniformly thin TEM specimen can be produced. For Zn, it was found that optimal results were attained if the next set of milling steps was performed while the sample surface was tilted into the beam at a ~14-deg angle. After the first side of the W line was milled, the sample was rotated so that the surface on the opposite side of the W line could be milled in an analogous manner. The sample was returned to the 0-deg tilt position, and a series of milling steps at a beam current of 250 pA were used to reduce the membrane thickness to ~1 μm. In order to cut the membrane free from the bulk, the sample was then tilted into the beam at an angle of ~60 deg. The 60-deg tilt permitted the Ga^+ beam to impinge on the lower portion of the specimen surface so that cuts could be made along the bottom edge and the lower two-thirds of the distance up one side of the specimen. These cuts made at 60 deg are part of the lift-out proceedure.[19] The FIB image in Figure 9, observed at this 60-deg tilt, reveals the thin W line, the grain morphology (*via* channeling contrast), and a hole in the upper left-hand corner of the specimen. The hole in the specimen face is a cross-sectional view of what is most likely a pre-existing pore. The grains imaged in the FIB are ~10 to 20 μm, which is consistent with those observed in the mechanically polished and etched particle shown in Figure 3(b). After once again returning to the 0-deg tilt position, the specimen thickness was further reduced to electron transparency (~100 nm) by milling alternating sides of the W line with a fine 63 pA beam. The left and right sides of the thin membrane were then milled away, thus freeing the specimen for lift out.

E. *Lift Out*

The ~5-μm wide by 20-μm long electron transparent membrane was extracted or lifted out from the bulk sample using a Narishige model MMO-202D three-axis hydraulic micromanipulator and was then deposited onto a 400-mesh carbon-coated copper TEM grid for subsequent TEM analysis. The lift-out technique has been described in detail elsewhere,[19] but the procedure is summarized below for completeness. The micromanipulator is used in conjunction with an LOM so that the FIB-milled TEM specimen can be located and observed as it is removed from the bulk. A glass rod that had been previously heated and pulled to a thin point of ~1 μm in diameter is attached to the robotic arm of the micromanipulator. The glass rod is first positioned close to the specimen using the coarse manual x, y, and z translational controls. Then, using the three-axis hydraulic micromanipulator, the tip of the glass rod is gently brought into contact with the top surface of the membrane, as shown in the schematic diagram in Figure 10. The TEM

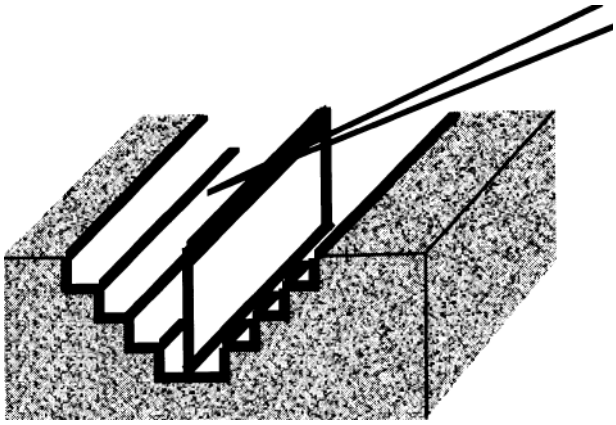


Fig. 10—A schematic diagram illustrating the lift out of the thin electron transparent membrane from the bulk sample.

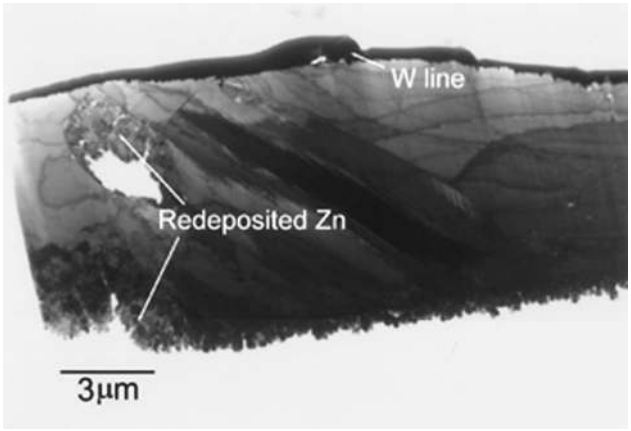


Fig. 11—A low-magnification BF TEM image of the thin membrane prepared by the FIB TEM lift-out technique.

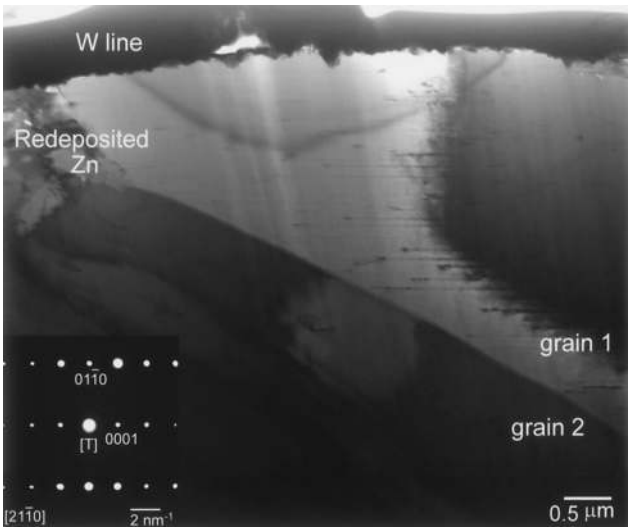


Fig. 12—A BF TEM image of the Zn powder and an SADP from grain 2.

specimen clings to the glass rod by what is believed to be electrostatic attraction and is then easily removed from the bulk material. After the membrane is free of the bulk, a TEM grid is moved into position under the glass rod, and the membrane is lowered and carefully placed onto one of the centrally located grid squares. At this point, the specimen is ready for insertion into the TEM. The total preparation time for this specimen, including mounting the powder, FIB milling, and insertion into the TEM, was roughly 3 hours. The total milling time was only 1 hour and 10 minutes. This represents a tremendous saving in specimen preparation time compared with other techniques.

F. *TEM Analysis*

The TEM analysis of the Zn lift-out specimen was performed using a PHILIPS* EM 430 TEM operating at 300 kV with a LaB_6 electron source. Figure 11 is a low-magnification, bright-field (BF) TEM image of the Zn membrane shown in Figure 9 after it was lifted out of the bulk and micromanipulated onto a TEM grid. The W line is visible at the top of the ~20-μm long by 5-μm wide electron transparent specimen. The hole that was noted in Figure 9 is still obvious. The ragged appearance on the bottom edge of the specimen is consistent with sputtered material that has been redeposited during FIB milling. The large grain size, consistent with the metallographically prepared sample shown in Figure 3(b) indicates that a specimen representative of the bulk microstructure was successfully prepared. It should be noted, however, that Ga^+ implantation was observed by energy dispersive spectroscopic analysis, and while the microstructure is preserved during FIB specimen preparation, the chemistry of the specimen may be altered.

*PHILIPS is a trademark of Philips Electronic Instruments Corp., Mahwah, NJ.

Figure 12 is a BF TEM image of the Zn lift-out specimen. The W line marks the top of the specimen, and the redeposited Zn in the upper left-hand corner borders the hole. A grain boundary within the Zn is evident in the micrograph. The ~10 to 20 μm grain size of the Zn lift-out specimen is sufficiently large to yield the $[2\bar{1}\bar{1}0]$ single-crystal selected area diffraction (SAD) pattern that was obtained from grain land is shown in the inset of the micrograph.

Perhaps the most important observation to be made from Figures 11 and 12 may be the absence of the mottled appearance characteristic of conventional Ar ion milling-induced dislocation loops. There is no evidence of milling damage even in the region closest to the W line that represents the zone of most intense Ga irradiation. In addition, previous work has shown that significant heating and redeposition of Zn onto the specimen surface may occur without the use of a liquid nitrogen (LN_2) cooling stage during conventional Ar ion milling.[37] The samples produced in this work were FIB milled at ambient temperatures without the use of a LN_2 cooling stage. This is yet another advantage to the use of the FIB TEM specimen preparation technique.

Figure 13 shows a higher magnification BF TEM micrograph of the region immediately surrounding the hole shown in Figures 9 and 11. The contrast variation in the image and the SAD ring pattern of the region indicate that the material located at the perimeter of the hole is fine grained (*i.e.*, <0.25 μm) polycrystalline Zn. This microstructure is inconsistent with the rest of the specimen but would be anticipated in a region where sputtered Zn has redeposited onto the specimen. Evidence suggests that the

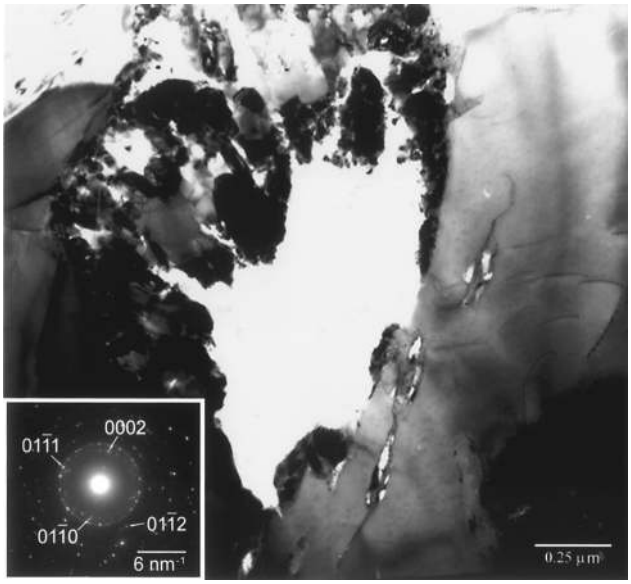


Fig. 13—A BF TEM image and corresponding SADP of the region surrounding a pore in the Zn powder. Note the presence of the fine-grained polycrystalline Zn, which is consistent with the redeposition of sputtered Zn onto the edges of the hole.

hole is actually a remnant of a pore that was part of the particle's original internal microstructure, as shown in Figures 2 and 3(a). The cross section of a pore is a concavity in the face of the sample,as shown in Figure 9. A concavity would act as a collection site or reservoir for redeposited sputtered material from the initial stages of milling. Thus, the concave region will tend to retain this sputtered material even after the final thinning stages.

III. CONCLUSIONS

The imaging and high resolution milling capabilities of the FIB have made it possible to preselect a region and mill a TEM specimen with submicrometer precision. Because thinning is uniform across the sample surface, large electron transparent regions on the order of 20×5 μm are made available for TEM observation. Conventional methods of specimen preparation can be very sample specific, and the development of a technique suited to a new material may be very time consuming. In contrast, it is now possible to take virtually any material or starting sample geometry with little or no prior sample preparation and produce a TEM specimen ready for analysis in 3 to 5 hours, as illustrated in the present work and in References 18 and 19.

We have shown that the FIB lift-out technique is viable for rapid production of site-specific, high-quality SEM and TEM samples of Zn powders. The microstructural features are consistent between samples prepared by FIB and those prepared by other techniques.

ACKNOWLEDGMENTS

This work was made possible through the generous support of Cirent Semiconductor and a Department of Defense NDSEG fellowship, Contract No. P-34862-RT-NDF. Special thanks to Suzi Orend, Larry Plew, and Cathy Vartuli, for their assistance, and to Jeff Bindell for his support of the University of Central Florida/Cirent Semiconductor partnership.

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