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4 **METHOD AND APPARATUS FOR RAPID SAMPLE PREPARATION IN A**
5 **FOCUSED ION BEAM MICROSCOPE**
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10 **Patent Application of**
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12

13 **CLAIM FOR PRIORITY**

14 This patent application claims priority from U.S. provisional patent application
15 No. 60/19,046, filed on November 11, 2003.

16 **TECHNICAL FIELD**

17 This disclosure relates to the use of focused ion-beam (FIB) microscopes for the
18 preparation of specimens for later analysis in the transmission electron microscope
19 (TEM), and apparatus to facilitate these activities.

20 **BACKGROUND**

21 The structural artifacts, and even some structural layers, in the device region
22 and interconnect stack of current integrated-circuit devices can be too small to be
23 reliably detected with the secondary electron imaging in a Scanning Electron
24 Microscope (SEM), or FIB, which offers a bulk surface imaging resolution of
25 approximately 3 nm. In comparison, TEM inspection offers much finer image
26 resolution (< 0.1 nm), but requires electron-transparent (< 100 nm thick) sections of the
27 sample mounted on 3 mm diameter grid disks.

28 Techniques were later developed for cutting out and removing specimens for
29 examination that required little or no preliminary mechanical preparation of the initial
30 semiconductor die sample before preparation in the FIB. These lift-out techniques
31 include an "ex-situ" method that is performed outside the FIB chamber, and "in-situ"
32 methods performed inside the FIB.

1 The in-situ lift-out technique is a series of FIB milling and sample-translation
2 steps used to produce a site-specific specimen for later observation in a TEM or other
3 analytical instrument. During in-situ lift-out, a sample of material (usually wedge-
4 shaped) containing the region of interest is first completely excised from the bulk
5 sample, such as a semiconductor wafer or die, using ion-beam milling in the FIB. This
6 sample is typically 10 x 5 x 5 μm in size. Removal of the lift-out sample is then
7 performed using an internal nano-manipulator in conjunction with the ion-beam assisted
8 chemical vapor deposition (CVD) process available with the FIB tool. A suitable nano-
9 manipulator system is the Omniprobe AutoProbe 200, manufactured by Omniprobe,
10 Inc., of Dallas, Texas. The material deposited in the CVD process is typically a metal or
11 an oxide.

12 A TEM sample holder is then positioned in the field of view in the FIB, and the
13 nano-manipulator is used to lower the lift-out sample onto the edge of the sample
14 holder. The CVD metal deposition capability inside the FIB vacuum chamber is then
15 used to secure the sample to the TEM sample holder. Once the sample is attached to the
16 TEM sample holder, the probe-tip point is separated from the sample by ion milling.
17 The portion of the method involving the operations that include the TEM sample holder
18 is referred to as the "holder attach" step. The sample may then be milled using
19 conventional FIB milling steps to prepare a thin area for TEM inspection or other
20 analysis. Details on methods of in-situ lift-out may be found in the specifications of U.S.
21 Patents Nos. 6,420,722 and 6,570,170. These patent specifications are incorporated into
22 this application by reference, but are not admitted to be prior art with respect to the
23 present application by their mention in this background section.

24 The in-situ lift-out technique has become popular, because this method allows
25 one to both take advantage of the FIB's unique capabilities and to extend these
26 capabilities to the inspection of structures and defects in next-generation devices. Due
27 to the small ion-beam spot size achievable by new FIB instruments (e.g., < 10 nm), FIB
28 specimen preparation techniques presently offer the best spatial resolution where site
29 specificity is required.

30 A variation of this in-situ lift-out process involves "backside milling" of the lift-
31 out sample. This variation was developed in response to the problem known as the
32 "shower curtain" effect, in which non-uniform high-density materials on the surface of

1 an integrated circuit produce a non-planar face on the lift-out sample after final thinning
2 for TEM preparation. These non-planar surfaces have vertical ridges, parallel to the ion
3 beam direction, that result from slower ion milling rates of denser materials near the top
4 of the sample, where the top is defined as the edge closest to the ion beam source. Such
5 non-uniform layers are quite common in integrated circuits, such as copper or aluminum
6 interconnect traces and tungsten contacts. A planar surface on the lift-out sample in the
7 area thinned for TEM inspection is important for TEM techniques such as electron
8 holography. Backside milling involves inverting the sample before final thinning, so
9 that high density materials in or near the active layer of the integrated circuit no longer
10 impact the result of ion milling.

11 The process of in-situ lift-out can be simplified into three successive steps. The
12 first is the excision of the sample using focused ion-beam milling and extraction of the
13 sample from its trench. The second is the "holder-attach" step, during which the sample
14 is translated on the probe-tip point to the TEM sample holder. Then it is attached to the
15 TEM sample holder (typically with ion beam-induced metal deposition) and later
16 detached from the probe-tip point. The third and final step is the thinning of the sample
17 into an electron-transparent thin section using focused ion beam milling.

18 A significant portion of the total time involved in completing a TEM sample
19 with in-situ lift-out is spent during the holder-attach step. The relative amount of time
20 involved depends on the amount of time required to mechanically isolate the lift-out
21 sample from the initial bulk sample (ion beam milling rate), but will vary between 30%
22 to 60% of the total time for TEM sample preparation. The elimination of the holder-
23 attach step provides several key revenue and resource-related advantages, due to the
24 elimination of the need to transfer and attach the TEM sample to the TEM sample
25 holder.

26 For example, without the holder-attach step the semiconductor wafer could be
27 returned to a process flow immediately after lift-out. Thinning of the sample can be
28 performed later in an off-line FIB. This reduces the load on the critical in-line (clean
29 room) FIB, which makes lift-out for process control more practical, and reduces the
30 level of expertise required by process engineers who operate the in-line FIB.

31 In order to eliminate the holder-attach step, the probe-tip point with the sample
32 attached can be directly joined to the material that will form the TEM sample holder by

1 a suitable method that will preserve the attachment between the lift-out sample and
2 probe-tip point, and prevent the probe-tip point and the sample from separating from
3 the sample holder during storage or inspection in the TEM. The assembly should not
4 interfere with the normal operation of the TEM or other intended analytical instrument,
5 and should survive well in the internal environment of the TEM, or other intended
6 analytical instrument. These suitable methods include, but are not limited to,
7 mechanical deformation of the sample holder material or probe-tip point material, or
8 both; electrical or thermal bonding (such as, electrical welding) of the probe-tip point to
9 the TEM sample holder material; bonding the probe-tip point to this material with a
10 suitable glue or adhesive; bonding the probe-tip point to the TEM sample holder
11 material with a CVD or evaporated material; or other suitable means. This direct
12 attachment of the probe-tip point with the sample attached to it, to the TEM sample
13 holder can be performed inside or outside the vacuum chamber of the FIB or other
14 analytical instrument.

15

SUMMARY

16 The preferred embodiment includes a coupon for preparing a TEM sample
17 holder. The coupon comprises a sheet of material, preferably copper or molybdenum,
18 and its surface may be planar or have corrugated structure. The sheet includes a TEM
19 sample holder form, and there is at least one section of the sheet connecting the TEM
20 sample holder form to other portions of the sheet.

21 In another preferred embodiment, a TEM sample holder form comprises a sheet
22 of material that has a C-shaped hole. The C-shaped hole defines the outer
23 circumference of a ring, and the mouth of the C-shaped hole defines a land of material.
24 The land connects the ring to the sheet, and there is a path across the sheet connecting
25 the C-shaped hole to the edge of the sheet.

26 The TEM sample holder comprises a ring, and the ring has a circumferential gap.
27 The circumferential gap is formed by cutting the circumferential gap from a TEM
28 sample holder form by pressing this form between two dies. The TEM sample holder
29 may further comprise one or more probe-tip points embedded in the ring, where every
30 probe-tip point further includes an attached sample. Probe-tip points may be embedded
31 in the ring by applying pressure to the ring and to one or more tip ends, so as to cause

1 plastic flow of the ring material around the shank, or they can be attached to the TEM
2 sample holder using adhesives, or by electrical or thermal welding techniques.

3 In another preferred embodiment, the TEM sample holder comprises a
4 rectangular or any other geometrical shape, which can be used for holding one or more
5 probe-tip points, every one of which comprises an attached sample.

6 A method for preparing a sample for examination in a TEM comprises the steps
7 of attaching a sample to the tip of a probe; joining the probe-tip point to a TEM sample
8 holder; and forming a TEM sample holder from the probe-tip point and the TEM
9 coupon.

10 The preferred embodiment further includes a press for cutting a TEM sample
11 holder from a TEM coupon and joining a probe-tip point with an attached sample to the
12 TEM sample holder. The press includes: an outer die; an inner die situated inside the
13 outer die; a former rod opposing the inner and outer dies; and, a shear punch situated
14 coaxially with the former rod. A hold-down spring biases the former rod toward the
15 inner die. A trigger or other mechanism responsive to the contact of the former rod and
16 the inner die, and an actuator responsive to the trigger, drive the shear punch toward the
17 inner and outer dies. This press can be located either inside or outside the vacuum
18 chamber of the FIB or other analytical instrument.

19 DRAWINGS

20 Figure 1 is a plan view of the TEM coupon of the preferred embodiment, where
21 the sample holder has a ring shape and one probe tip is intended for use.

22 Figure 2 is a plan view of the TEM coupon of Figure 1, showing a probe tip
23 positioned across it before embedding and cutting.

24 Figure 3 is a plan view of the TEM coupon of an alternate embodiment, where
25 the sample holder has a ring shape and four probe tips are intended for use.

26 Figure 4 is a plan view of the TEM sample holder and probe-tip combination,
27 formed from the TEM coupon of Figure 1, with a ring opening allowing FIB ion milling
28 of the top surface of the lift-out sample.

29 Figure 5 is a plan view of the TEM sample holder and the four probe-tip
30 combination, formed from the TEM coupon of Figure 2, with a ring opening allowing
31 FIB ion milling of the top surface of the lift-out sample.

1 Figure 6 is a cross-sectional view of a probe-tip point embedded in a TEM
2 coupon.

3 Figure 7 is a cross-sectional view of a probe-tip point attached to a TEM
4 corrugated coupon via electrical or thermal bonding.

5 Figure 8 is a cross-sectional view of a probe-tip point attached to a TEM sample
6 holder using adhesive.

7 Figure 9 is a cross-sectional view of a probe-tip point bonded to the TEM sample
8 holder material with a CVD or evaporated material.

9 Figure 10 is a partial cross-sectional view of the press and the shear punch of the
10 preferred embodiment.

11 Figure 11 is an enlarged partial cross-sectional view of the press and the shear
12 punch of Figure 10.

13 Figure 12 is a perspective view of the shear punch of Figure 10 and the inner and
14 outer dies.

15 Figure 13 is a perspective view of the shear punch of Figure 10, shown engaging
16 a coupon and a probe tip.

17 Figure 14 is a transverse view of the probe-tip point, tip former rod of the
18 preferred embodiment positioned above a probe-tip point, and a TEM sample holder,
19 with the probe-tip point embedded in a TEM sample holder.

20 Figure 15 is a plan view of the TEM sample holder and probe-tip point
21 combination shown in Figure 4, with the circumferential gap made in the sample holder
22 ring for the backside milling.

23 Figure 16 is a plan view of the TEM sample holder and four probe-tip points
24 combination shown in Figure 5, with the ring opening made for the backside milling.

25 Figure 17 is a transverse view of a corrugated TEM coupon.

26 Figure 18 is a transverse view of a probe-tip point, embedded in a corrugated
27 TEM coupon.

28 Figure 19 is a plan view of the TEM coupon of the preferred embodiment, where
29 the sample holder has a rectangular shape and four probe tips are intended for use, with
30 an opening allowing the sample top surface FIB ion milling.

31 Figure 20 is a plan view of the TEM coupon of the preferred embodiment, where
32 the sample holder has a rectangular shape and four probe tips are intended for use, with

1 an opening allowing backside FIB ion milling of the bottom surface of lift-out samples.

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DISCLOSURE

4 The preferred embodiment includes a novel method and apparatus for adjoining
5 a probe tip with attached sample to a TEM sample holder that replaces the holder-attach
6 step of the conventional method. In the preferred embodiment, this mechanical process
7 is performed outside the vacuum chamber, although it could be performed inside the
8 FIB chamber as well. In the preferred embodiment, the first step of the in-situ lift-out
9 procedure (the excision of the sample) is completed in the FIB, and the probe-tip point
10 with the sample attached is then removed from the FIB chamber. This removal can be
11 accomplished by a number of means, including but not limited to, removal of the probe
12 tip and attached sample through the sample door of a FIB equipped with a door,
13 translation of the probe tip and attached sample through a vacuum airlock on the nano-
14 manipulator device, or the translation of the probe-tip point and attached sample in a
15 cassette that passes through a vacuum airlock on the FIB chamber. All but the first
16 means listed do not require that the FIB vacuum chamber be vented to atmosphere,
17 which offers cycle time reduction and long-term equipment reliability advantages.

18

The Coupon

19 In the preferred embodiment, the probe-tip point (160) of a nano-manipulator
20 probe (150) is attached to a TEM coupon (100) by a combined mechanical forming and
21 cutting operation. As shown in Figure 1, the TEM coupon (100) is a sheet of material of
22 approximately the same thickness as the final sample holder (170). The TEM coupon
23 (100) contains the shape of the final sample holder (170) (the "TEM pre-form"),
24 although this pre-form has not yet been completely mechanically isolated. Most of the
25 final shape of the typical 3 mm TEM sample holder (170) can be created in the sheet in
26 advance, as consumable coupons (100). The pre-form is still attached to the coupon
27 (100) with tabs, lands, or other sections of the sample holder material (120). The pre-
28 form has a ring (180) that will be a part of the final TEM sample holder (170). The ring
29 (180) is thus defined by a C-shaped hole (135) in the coupon (100). The mouth of the
30 C-shaped hole (135) is the attaching land (120). Other enclosing shapes, such as
31 rectangles, may also be used.

1 The holder material is preferably soft copper, but may also be molybdenum,
2 aluminum, gold, silver, nickel or beryllium, if appropriate to the application. The
3 coupon (100) orients the sample holder form (170) and holds it in place during the
4 mechanical steps of the isolation process, described below. Figure 2 shows a nano-
5 manipulator probe tip (150) placed across the coupon (100). The probe (150) has a
6 probe-tip point (160) that holds a sample (140) for analysis. Typically, the probe-tip
7 point (160) is a fine tungsten needle.

8 The TEM coupon (100) may also be fabricated from a material harder than
9 copper, such as molybdenum or it may have a surface structure that facilitates the
10 mechanical embedding of the probe-tip point (160) in the coupon material. A good
11 example is a surface structure with corrugations (175) that have a period approximately
12 the same or less than the probe-tip point (160) diameter. Figures 7 and 17 show cross-
13 sections of a corrugated structure. In Fig. 7, the corrugation period is about half the
14 diameter of the probe-tip point (160). The corrugations (175) may be periodic, such as
15 continuous rows or ridges roughly aligned in the direction of the probe-tip point, rows of
16 individual posts, or non-periodic free-form elevations. These structures can be easily
17 deformed to lock the probe-tip point (160) in place.

18 The remaining tabs, or lands (120) of the coupon material, which connect the
19 partially formed TEM sample holder (170) to the coupon (100), are severed during the
20 combined mechanical forming and cutting operation, described below. The TEM
21 sample holder (170) is preferably produced in the shape of a ring (180) with a
22 circumferential gap (190) to enable later FIB ion milling of either top or bottom surface
23 of the sample (140) in the plane of the TEM sample holder (170), thus producing an
24 electron-transparent thin section that would be approximately parallel to the plane of the
25 TEM sample holder (170). Other shapes that allow for a circumferential gap (190) in
26 the ring (180) of the formed TEM sample holder (170) may also be used. Figs. 19 and
27 20, for example, shows a TEM sample holder (170) having two gaps (190), where the
28 shape of the TEM sample holder (170) is rectangular.

29 Figs. 4, 5, and 19 show TEM sample holders (170) with probe-tip points (160)
30 mounted for top-side ion milling of samples (140). Figs. 16 and 20 show TEM sample
31 holders (170) with probe-tip points (160) mounted for back-side milling of samples
32 (140).

Methods of Forming the TEM Sample Holder

2 The probe-tip point (160) with the sample (140) attached can be joined to the
3 material that will form the TEM sample holder (170), so as to preserve the attachment
4 between the sample (140) and the probe-tip point (160), and prevent the probe-tip point
5 (160) and sample (140) from separating from the TEM sample holder (170) during
6 transportation, storage or inspection in the TEM. The assembly should not interfere
7 with the normal operation of the TEM, or other intended analytical instrument, and
8 should survive well in the internal environment of the TEM, or other intended analytical
9 instrument.

Figures 6-9 and 18 show methods for joining the probe-tip point (160) to the TEM coupon (100). Figure 6 is a cut-away view of mechanical deformation of the material of the coupon (100) or probe-tip point (160), or both. Figure 7 depicts electrical or thermal bonding (320), such as welding, of the probe-tip point (160) to the coupon (100). Figure 7 also shows corrugations (175) in the TEM sample holder material; in this case the corrugation period is about the same as the diameter of the probe-tip point (160). Figure 8 shows bonding the probe-tip point (160) to the TEM sample holder (170) material with a suitable glue or adhesive (330). Figure 9 shows bonding the probe-tip point (160) to the TEM sample holder (170) material with a CVD or evaporated material (340).

Once the TEM sample holder (170) with one or more probe-tip points (160) with samples (140) attached to it has been created, it can be returned to the FIB for the final thinning operation, during which the desired portion of the lift-out sample (140) or samples is thinned to electron transparency (typically 50-250 nm). This final thinning can be performed in an off-line FIB to maximize throughput of the in-line FIB and to take advantage of the efficiency, expertise and dedicated resources of the off-line FIB lab that can be located outside the clean room. However, if the apparatus for attaching a sample to a TEM sample holder is located inside the FIB, the final thinning operation can be performed immediately.

In an alternative method, the final thinning step can be performed in the FIB after the lift-out step and before the probe-tip point (160) with the sample (140) attached is removed from the FIB for attachment to the TEM sample holder outside the FIB. In this method, it is not required to return the mechanically formed TEM sample holder

1 (170) with the sample (140) attached, to the FIB for final thinning. However, the final
2 thinning process requires the additional time in the initial FIB. In this method, the
3 probe-tip point (160) with the sample (140) attached is translated to a suitable location
4 in the FIB, and the ion beam in the FIB is then used to perform the final thinning step.
5 Then, the probe-tip point (160) with the thinned sample (140) attached is removed from
6 the FIB and attached to the TEM sample holder (170) using the mechanical forming and
7 cutting process described above. It is recommended, but not required, to stabilize the
8 probe-tip point (160) mechanically to reduce any vibration in the probe-tip point (160)
9 relative to the FIB chamber to an acceptable level, or to reduce any mechanical drift of
10 the probe-tip point (160) relative to the FIB chamber. The probe-tip point (160) with
11 the sample (140) attached can be mechanically stabilized by making mechanical contact
12 between the probe-tip point (160) and a suitably stable surface in the FIB, or between
13 the sample (140) and a suitable surface or object in the FIB. For example, the edge or a
14 corner of a mechanical structure attached to the FIB stage, and the probe-tip point (160)
15 can be brought together into mechanical contact. Or, the bottom edge of the sample
16 (140) can be brought into mechanical contact with the surface of the sample stage or any
17 stable mechanical object attached to the sample stage (e.g., the surface of the wafer).
18 The stable object can be rigid, or can be deformable by plastic or elastic deformation, to
19 accept the shape of the probe-tip point (160) or lift-out sample (140) and further dampen
20 any relative mechanical vibration in the probe-tip point (160).

21 In another alternative method, the final thinning step can be performed in the
22 FIB after the lift-out step and before the probe-tip point (160) with sample (140)
23 attached is joined to the TEM sample holder inside the FIB vacuum chamber. In this
24 method, the probe-tip point (160) with the lift-out sample (140) attached is translated to
25 a suitable location in the FIB, and the ion beam in the FIB is then used to perform the
26 final thinning step. Then, the probe-tip point (160) with the thinned sample (140) can
27 be attached to the TEM sample holder (170) inside the FIB vacuum chamber using the
28 mechanical forming and cutting process described above. In this method, the apparatus
29 for attaching a sample to a TEM sample holder is located inside the same FIB vacuum
30 chamber. Hence, the in-situ lift-out, the attachment of a probe-tip point with a sample
31 attached to it to a TEM sample holder, and the final thinning operation can be performed
32 as steps of one process inside the FIB vacuum chamber.

Sample Holder Forming Apparatus

Figures 1 and 2 show a TEM coupon (100), as described above. The land (120) that connects the sample holder portion (170) of the coupon (100) to the rest of the coupon (100) will be severed to form the TEM sample holder (170) during the cutting and forming operation. The thickness of the coupon (100) is determined by the thickness required to embed and mechanically lock the probe-tip point (160) in the coupon (100) material and still provide for sufficient mechanical strength of the final sample holder (170) to prevent unwanted folding or separation of the TEM sample holder (170) at the probe-tip point (160) embedding location. For example, for the case of a 125 μm (.005") diameter tungsten probe-tip point (160), a thickness of 250-500 μm (.010"-.020") of copper is appropriate for the coupon (100). Both the sample holder (170) material and the surrounding coupon (100) material are slightly recessed in a probe-tip point cut-off zone (130) to allow space for the cutting surfaces to cut the probe-tip point (160) without leaving any portion of the severed probe-tip point (160) extending beyond the 3 mm outside diameter of a standard TEM sample holder (170) or extending beyond the outside border of a standard TEM sample holder (170) of any other suitable shape.

Alignment holes (110) are included to permit alignment of the coupon (100) in the mechanical apparatus that performs the cutting and forming operation. In the case of a C-shaped TEM sample holder (170), the probe-tip point clearance slot (125) (Figure 3) is a straight slot through the coupon (100), radiating outward from the center of the TEM sample holder (170) beyond the outer diameter of the TEM sample holder (170), that provides clearance for the probe-tip point (160) to permit alignment of the probe-tip point (160) along the surface of the TEM sample holder (170) before the cutting and forming operation.

During the cutting and forming operation, a TEM sample holder (170) is cut from the coupon (100) (Figure 4). As discussed above, the TEM sample holder (170) can be produced in a C-shape form, or other shape having a circumferential gap (190) to enable later FIB ion milling of the lift-out sample in the plane of the TEM sample holder (170) to produce an electron-transparent thin section approximately parallel to the TEM sample holder (170) plane, or in any other shape allowing the same process. For later milling of the top surface of a sample (140), the gap (190) can be cut from the form at

1 the mouth of the C-shaped hole (135), defined by the land (120) connecting the form to
2 the coupon (100). For later milling of the bottom surface of a sample (140), the gap can
3 be cut from the form at a location approximately opposite the mouth of the hole (135).

4 During the cutting and forming operation, the harder tungsten probe-tip point
5 (160) is pressed into the softer material of the TEM sample holder (170), and the portion
6 of the probe-tip point (160), extending outside the outer diameter of the 3 mm TEM
7 sample holder shape (170), is cut off. The TEM sample holder (170) material is
8 induced to plastically deform so that the copper material mechanically surrounds the
9 probe-tip point (160) to lock it in place (Figure 6).

10 Figures 10-13 show a typical process for the cutting and forming operation. The
11 operator places the TEM coupon (100) on the outer die (280) (this operation can be
12 performed by hand, if this operation is performed outside the FIB, or automatically, if it
13 is performed inside the same FIB vacuum chamber) and aligns every probe-tip point
14 (160) in such a way, that every probe-tip point (160) is aligned with the probe-tip point
15 clearance slot (125), and the sample (140), attached to a probe-tip point (160), is
16 oriented parallel to the plane of the TEM sample holder (170). The inner die (290) and
17 the outer die (280) both support the sample holder (170) and the probe-tip point (160).
18 Once every probe tip is secured, the operator positions by hand or automatically the
19 probe tip or probe tips, TEM coupon (100), and all supporting hardware under the main
20 mounting block (220), and actuates a pneumatic switch (310), causing the main
21 mounting block (220) and attached hardware to travel downward under the action of an
22 actuator (300) located above the main mounting block (220). The actuator (300) is
23 preferably pneumatic, but hydraulic or electrical actuators may also be used. There is
24 also the exhaust line (305) for pneumatic actuators.

25 Figures 11-13 show the forming and cutting operation as the main mounting
26 block (220) moves downwards. The former rod (250) contacts every probe-tip point
27 (160) and presses it down into the TEM sample holder (170) material. This continues
28 until the TEM sample holder and the probe-tip point interface build up enough
29 resistance to overcome the force of the hold down spring (230). The hold down spring
30 (230) force is set with a spring adjustment screw (240) to the desired force to ensure that
31 every probe-tip point (160) is pressed fully into the TEM coupon (100). The former rod

1 (250) includes one or more teeth (260) that flow the holder material around the probe-tip
2 point (160) encasing it as it is pressed down (Figure 14).

3 Once resistance to the spring (230) is overcome and the former rod (250)
4 movement is stopped, the shear punch (270) continues its travel downward, using the
5 support of both the inner die (290) and the outer die (280) to shear every probe-tip point
6 (160) at the desired length, sever the tab (120) connecting the TEM sample holder (170)
7 from the rest of the TEM coupon (100) and create the C-shaped opening, or the opening
8 of any other suitable shape, in the holder (170). The operator then releases a pneumatic
9 switch to return the main mounting block (220) and attached hardware to its original
10 position, leaving the TEM sample holder (170) separated from the TEM coupon (100)
11 and containing one or more probe-tip points (160) with the samples (140) attached.

12 Since those skilled in the art can modify the specific embodiments described
13 above, I intend that the claims be interpreted to cover such modifications and
14 equivalents.

15 I claim:

CLAIMS

- 1
2
- 3 1. A coupon for preparing a TEM sample holder, the coupon comprising:
4 a sheet of material,
5 the sheet comprising a TEM sample holder form; and,
6 one or more paths through the sheet connecting the TEM sample holder form to the edge
7 of the sheet.
8
- 9 2. The coupon of claim 1 where the sheet comprises a metal selected from the
10 group consisting of copper, molybdenum, aluminum, gold, silver, nickel and beryllium.
11
- 12 3. The coupon of claim 1 where the sheet has surface corrugations.
13
- 14 4. The coupon of claim 1 where the coupon further comprises alignment holes, for
15 aligning the coupon in a press.
16
- 17 5. The coupon of claim 1 further comprising:
18 at least one hole in the coupon defining the outer boundary of a TEM sample
19 holder form; the hole having a mouth;
20 the mouth of the hole defining a land of material;
21 the land connecting the TEM sample holder form to the sheet; and,
22 one or more paths through the sheet connecting the hole to the edge of the sheet.
23
- 24 6. The coupon of claim 5 where the hole has a C-shape.
25
- 26 7. The coupon of claim 5 where the TEM sample holder has a rectangular shape.
27
- 28 8. The coupon of claim 5 where the hole has a rectangular shape.
29
- 30 9. A TEM sample holder, the TEM sample holder comprising:
31 a ring, the ring having a circumferential gap;

1 the circumferential gap formed by cutting the circumferential gap from a TEM
2 sample holder form.

3

4 10. The TEM sample holder of claim 9, where the ring is defined by a hole in a
5 coupon for preparing a TEM sample holder; the hole having a mouth; where the gap is
6 formed by cutting the circumferential gap from a TEM sample holder form at the mouth
7 of the hole, so as to allow FIB milling of the top surface of a sample.

8

9 11. The TEM sample holder of claim 9, where the ring is defined by a hole in a
10 coupon for preparing a TEM sample holder; the hole having a mouth; where the gap is
11 formed by cutting the circumferential gap from a TEM sample holder form at a location
12 approximately opposite the mouth of the hole, so as to allow FIB milling of the bottom
13 surface of a sample

14

15 12. The TEM sample holder form of claim 9, where the cutting operation comprises
16 pressing the TEM sample holder form between two dies.

17

18 13. The TEM sample holder of claim 9, further comprising:
19 one or more probe-tip points embedded in the ring; and,
20 one or more samples attached to the probe-tip points.

21

22 14. The TEM sample holder of claim 13, where the probe-tip points are embedded in
23 the ring by applying pressure to the ring and the probe-tip points so as to cause plastic
24 flow of the ring about the probe-tip points.

25

26 15. A method of preparing a sample for examination in a TEM, the method
27 comprising:

28 attaching a specimen to the point of a probe tip;
29 joining the probe-tip point to a TEM sample holder coupon;
30 forming a TEM sample holder from the probe-tip point and the TEM coupon.

31

1 16. The method of claim 15 where the sample is prepared for the TEM examination
2 outside the FIB vacuum chamber.

3

4 17. The method of claim 15 where the sample is prepared for the TEM examination
5 inside the in-line FIB vacuum chamber.

6

7 18. The method of claim 15 where the step of joining one or more probe-tip points
8 to the TEM coupon and forming a TEM sample holder further comprises:

9 providing a TEM sample holder form in the TEM coupon;

10 orienting the probe-tip points and the TEM coupon so that the specimen is
11 approximately centered in the sample holder;

12 embedding the probe-tip points in the TEM sample holder form;

13 cutting off that portion of every probe-tip point outside the TEM sample holder
14 form boundary; and,

15 cutting a TEM sample holder free from the TEM coupon.

16

17 19. The method of claim 18 where the step of embedding further comprises applying
18 pressure to the TEM sample holder and the probe-tip points so as to cause plastic flow
19 of the TEM sample holder material about the probe-tip points.

20

21 20. The method of claim 15 where the step of joining further comprises welding the
22 probe-tip points to the TEM sample holder.

23

24 21. The method of claim 15 where the step of joining further comprises attaching the
25 probe-tip points to the TEM sample holder with adhesive.

26

27 22. The method of claim 15 where the step of joining further comprises attaching the
28 probe-tip points to the TEM sample holder by chemical-vapor deposition.

29

30 23. The method of claim 18 where the cutting off of the portion of the probe-tip
31 points outside the TEM sample holder boundary and the cutting free of a TEM sample
32 holder are simultaneous.

1

2 24. A method of preparing a sample for examination in a TEM, the method
3 comprising:

- 4 providing one or more probe tips; the probe tips having a tip point;
- 5 excising a sample from a substrate inside a FIB;
- 6 attaching the sample to the probe-tip point inside the FIB;
- 7 removing every probe-tip point and a sample attached to it from the FIB;
- 8 joining every probe-tip point to a TEM coupon outside the FIB; and,
- 9 forming a TEM sample holder from the probe-tip point and the TEM coupon.

10

11 25. A method of preparing a sample for examination in a TEM, the method
12 comprising:

- 13 providing one or more probe tips; the probe tips having a tip point;
- 14 excising a sample from a substrate inside a FIB;
- 15 attaching the sample to the probe-tip point inside the FIB;
- 16 removing every probe-tip point and a sample attached to it from the FIB;
- 17 joining every probe-tip point to a TEM coupon inside the FIB; and,
- 18 forming a TEM sample holder from the probe-tip point and the TEM coupon
19 inside the FIB vacuum chamber.

20

21 26. The method of claim 24, where the step of joining the probe-tip point to the
22 TEM coupon and forming a TEM sample holder further comprises:

- 23 providing a TEM sample holder form in the TEM coupon;
- 24 orienting the probe-tip point and the TEM coupon so that the specimen is
25 approximately centered in the TEM sample holder;
- 26 embedding the probe-tip point in the TEM sample holder form;
- 27 cutting off that portion of the probe-tip point outside the TEM sample holder
28 form outer boundary; and,
- 29 cutting a TEM sample holder free from the TEM coupon.

30

1 27. The method of claim 26, where the step of embedding further comprises
2 applying pressure to the TEM sample holder and the probe-tip point so as to cause
3 plastic flow of the TEM sample holder material about the probe-tip point.

4

5 28. The method of claim 24, where the step of joining further comprises welding the
6 probe-tip point to the TEM sample holder.

7

8 29. The method of claim 24, where the step of joining further comprises attaching
9 the probe-tip point to the TEM sample holder with adhesive.

10

11 30. The method of claim 24, where the step of joining further comprises attaching
12 the probe-tip point to the TEM sample holder by chemical-vapor deposition.

13

14 31. The method of claim 24 where the cutting off of the portion of the probe-tip
15 point outside the TEM sample holder boundary and the cutting free of a TEM sample
16 holder are simultaneous.

17

18 32. The method of claim 24 further comprising returning the specimen to the FIB for
19 further thinning after forming the TEM sample holder.

20

21 33. A press for cutting a TEM sample holder from a TEM coupon and joining a
22 probe-tip point with a sample attached to it to the TEM sample holder, the press
23 comprising:

24 an outer die;

25 an inner die situated inside the outer die;

26 a former rod opposing the inner and outer dies;

27 a shear punch situated coaxially with the former rod;

28 a hold-down spring biasing the former rod toward the inner die; and,

29 an actuator for driving the shear punch toward the inner and outer dies.

30

31 34. The press of claim 33 where the former rod has teeth for flowing TEM sample
32 holder material around the probe-tip point.

1

2 35. A kit for preparing TEM sample holders, the kit comprising:

3 at least one TEM coupon;

4 at least one probe tip, the probe tip having a probe-tip point; and,

5 a press, for joining every probe-tip point to the TEM coupon and for cutting a

6 TEM sample holder from the coupon.

7

8 36. The kit of claim 35, where the coupon further comprises:

9 a sheet of material; and,

10 one or more paths through the sheet connecting the TEM sample holder form to

11 the edge of the sheet.

12

13 37. The kit of claim 35, where the sheet comprises a metal, selected from the group

14 consisting of copper, molybdenum, gold, silver, nickel and beryllium.

15

16 38. The kit of claim 35 where the sheet has surface corrugations.

17

18 39. The kit of claim 35 where the coupon further comprises alignment holes, for

19 aligning the coupon in a press.

20

21 40. The kit of claim 35 further comprising:

22 at least one hole in the coupon defining the outer boundary of a TEM sample

23 holder form; the hole having a mouth;

24 the mouth of the hole defining a land of material;

25 the land connecting the TEM sample holder form to the sheet; and,

26 at least one path through the sheet connecting the hole to the edge of the sheet.

27

28 41. The kit of claim 40, where the hole has a C-shape.

29

30 42. The kit of claim 40, where the TEM sample holder has a rectangular shape.

31

32 43. The kit of claim 35, where the press further comprises:

- 1 an outer die;
 - 2 an inner die situated inside the outer die;
 - 3 a former rod opposing the inner and outer dies;
 - 4 a shear punch situated coaxially with the former rod;
 - 5 a hold-down spring biasing the former rod toward the inner die; and,
 - 6 an actuator for driving the shear punch toward the inner and outer dies.
 - 7
- 8 44. The kit of claim 43, where the former rod has teeth for flowing the TEM sample
9 holder material around every probe-tip point.