

METHOD OF TRANSMISSION ELECTRON MICROSCOPE FOIL PREPARATION USING LASER

CROSS-REFERENCE TO RELATED APPLICATION

[0001] This application claims the benefit of priority to the Singapore application no. 10202205333W filed 20 May 2022, the contents of which are incorporated herein by reference in their entirety for all purposes.

TECHNICAL FIELD

[0002] Various embodiments of this disclosure relate to a method of preparing samples for transmission electron microscopy and to samples prepared by the method.

BACKGROUND

[0003] Transmission electron microscopy (TEM) is a useful way of examining internal structures of materials/products/devices in a broad range of industrial and scientific research activities, including in the manufacturing of semiconductors, optics, electronics, etc. Conventional methods of sample preparation for TEM analysis (such as mechanical grinding and ion milling) are known to be time consuming. Among the various methods of TEM sample preparation, focused ion beam milling is generally selected for preparing a sample at a known location because it can be more site-specific. Ion milling may cause direct damage at the surface of the sample, e.g., the sputtered materials may become re-deposited on the surface of the sample. Some ion species used in ion milling may also alter the physical properties of materials in the sample and may therefore be unsuitable for certain applications such as the TEM analysis of semiconductors.

SUMMARY

[0004] In one aspect, the present application discloses a method of preparing a sample for TEM analysis. The method includes thinning a specimen to a bulk thickness that is non-

transparent to electrons, the thinning being performed on a first surface of the specimen exclusively by laser milling using a laser. The method includes ablating a region in the first surface of the specimen such that the region is thinner than the bulk thickness, the ablating being performed exclusively by the laser within the region to leave other parts of the first surface unmodified by the ablating; and ending the ablating in response to sensing that at least one penetrated part of the region is transparent to electrons, such that a sampling point for the TEM analysis is selectable from an ablated area at or neighboring the at least one penetrated part. The method may include providing the laser at an angular displacement relative to a normal axis after the thinning and before the ablating such that the laser is directed at the specimen at an inclination angle during the ablating, in which the laser is directed at the specimen along the normal axis during the thinning. The method may include selecting a selected plurality of the region in the first surface; and repeating the ablating and the ending thereof for each of the selected plurality of the region.

[0005] In another aspect, the present application discloses a sample for TEM analysis prepared by the method disclosed herein, in which the sample includes one or more regions each forming a respective recess in a first surface, the respective recess being characterized by a non-uniform thickness along an inclined surface extending from the first surface to an ablated area neighboring a penetrated part, in which the penetrated part is characterized by a sampling thickness that is transparent to electrons.

[0006] In another aspect, the present application discloses a method of preparing a foil of a material. The method including thinning a specimen to a bulk thickness, the thinning being performed on a first surface of the specimen exclusively by laser milling using a laser; ablating a first region in the first surface of the specimen such that the first region is thinner than the bulk thickness, the ablating being performed exclusively by the laser; ending the ablating in response to sensing that at least one penetrated part of the first region is at or thinner than a

target thickness at which the material is transparent to electrons; and repeating the ablating and the ending thereof at another region contiguous with the first region until a predetermined contiguous area of the first surface is transparent to electrons.

[0007] In yet another aspect, the present application discloses a foil formed according to the above method, in which the foil includes a predetermined contiguous area having a foil thickness that is transparent to electrons. The foil thickness may be 50 nm or thinner than 50 nm.

BRIEF DESCRIPTION OF THE DRAWINGS

[0008] Various embodiments of the present disclosure are described herein with reference to the following drawings:

FIG. 1 is a schematic diagram of a system for preparing a sample according to one embodiment of the present disclosure;

FIGS. 2A and 2B are schematic diagrams illustrating different exemplary configurations of the helical optics of the system of FIG. 1;

FIG. 3 is a schematic diagram illustrating the method according to various embodiments of the present disclosure;

FIGS. 4A to 4F are schematic diagrams illustrating a specimen at various stages of preparation;

FIG. 5A is a schematic diagram illustrating a specimen undergoing ablating by a laser;

FIG. 5B is a magnified partial view of FIG. 5A;

FIGS. 6A to 6C are perspective views illustrating exemplary laser scanning paths of various embodiments of the present disclosure;

FIGS. 7A to 7C are cross-sectional views of TEM samples according to embodiments of the present disclosure; and

FIG. 8 illustrates a sectional perspective view of a TEM sample according to another embodiment of the present disclosure.

DETAILED DESCRIPTION

[0009] The following detailed description refers to the accompanying drawings that show, by way of illustration, various details, and embodiments of the present application. Modifications may be made to the examples described without departing from the scope of the invention as claimed. Features that are described in the context of one embodiment may correspondingly be applicable to the same or similar features in the other embodiments. Features that are described in the context of an embodiment may correspondingly be applicable to the other embodiments, even if not explicitly described in these other embodiments. Furthermore, additions and/or combinations and/or alternatives as described for a feature in the context of an embodiment may correspondingly be applicable to the same or similar feature in the other embodiments.

[0010] In the context of various embodiments, the articles “a”, “an” and “the” as used with regard to a feature or element include a reference to one or more of the features or elements. In the context of various embodiments, the term “about” or “approximately” as applied to a numeric value encompasses the exact value and a reasonable variance, e.g., within 10% of the specified value. As used herein, the term “and/or” includes any and all combinations of one or more of the associated listed items.

[0011] By “comprising” it is meant including, but not limited to, whatever follows the word “comprising”. Thus, use of the term “comprising” indicates that the listed elements are required or mandatory, but that other elements are optional and may or may not be present.

[0012] By “consisting of” is meant including, and limited to, whatever follows the phrase “consisting of”. Thus, the phrase “consisting of” indicates that the listed elements are required or mandatory, and that no other elements may be present.

[0013] FIG. 1 is a schematic diagram of one exemplary system 100 which can be used to perform various embodiments of the methods described in the present application. In this

example, the system 100 includes a laser source 104 and helical optics 106 (collectively, the laser 1040). For the sake of brevity, the term "laser" may refer to the hardware and/or the laser beam itself, as will be understood from the context by one skilled in the art. The laser 1040 is coupled to robotic support and controllable by a computing device or controller 102 such that the delivery of the laser and various laser parameters such as scanning speed, scanning direction, etc., are controllable via signal communication with the controller 102. The system 100 includes a sensor 170 which is preferably a photosensor. In a set-up in which a workpiece or a specimen 110 is supported on a platform with an upper surface of the specimen being presented to the laser, the sensor 170 is preferably disposed at a lower surface of the specimen. In a specimen 110 having a first surface 1182 and a second surface 1184 (e.g., see FIG. 4C), the second surface 1184 being a major and opposing surface facing away from the first surface 1182, if the first surface 1182 is oriented proximal to the laser 1040, the sensor 170 is preferably located at the second surface 1184 distal to the laser 1040.

[0014] The wavelength of the laser 1040 emitted from the laser source 104 may be within a range from, but is not limited to, extreme ultraviolet to far infrared. The pulse duration of the laser 1040 may be within a range from, but is not limited to, 10 femtoseconds to 10 picoseconds. The laser 1040 is configured to be operable for removing material from a workpiece or for ablation of a workpiece at a controllable ablation rate. As used herein, the term "ablation rate" refers to the amount of materials removed from the specimen by the laser in a unit of time. The system 100 may be configured to operate under vacuum or ambient atmosphere. For example, the system 100 is preferably operated under vacuum conditions if the laser 1040 has a wavelength in the range of ultraviolet radiation.

[0015] The helical optics 106 are configured to direct the laser 1040 toward the specimen 110 at an incident angle or an incident position controllable by the controller 102. Exemplary configurations of the helical optics 106 are illustrated schematically in FIGS. 2A and 2B. The

helical optics 106 may include wedge lenses 1062, 1064 and a focus lens 1066 accommodated in a housing 1068. The wedge lenses 1062, 1064 and the focus lens 1066 are aligned along a normal axis 1060 of the helical optics 106. Each of the wedge lenses 1062, 1064 is configured to be rotatable about the normal axis 1060. For example, each of the wedge lenses 1062, 1064 can be configured to rotate about the normal axis 1060 along the direction 1060a. Each of the wedge lenses 1062, 1064 may be further configured to be movable with respect to the housing 1068 along the normal axis 1060. For example, the wedge lenses 1062 and 1064 may be repositioned by displacing in the directions 1062a and 1064a with respect to the housing 1068, respectively.

[0016] In operation, the laser 1040 emitted from the laser source 104 travels into the helical optics 106 substantially parallel with the normal axis 1060. The laser 1040 is then directed / refracted by the wedge lens 1062, 1064 and the focus lens 1066, respectively. The helical optics 106, including the wedge lenses 1062, 1064 and focus lens 1066, are configured to direct the laser 1040 to radiate on the specimen 110 at a desired incident angle and desired position. In a first example, as is shown in FIG. 2A, the laser 1040 radiates on a surface of the specimen 110 at an incident angle β_1 and at a position spaced a distance d_1 from a center of the specimen 110. In a second example, as is shown in FIG. 2B, compared with the first example of Fig. 2A, the wedge lenses 1062 and 1064 are spaced further from each other, resulting in an incident angle β_2 that is different from β_1 . The laser 1040 is also repositioned at a position spaced apart from the center of the specimen by a distance d_2 different from d_1 .

[0017] Advantageously, the present method of preparing a sample for TEM analysis can be applied to any specimen or materials. This can be particularly useful in applications where one is concerned that conventional ion milling may modify the material composition of the specimen. The specimen may include metallic materials, inorganic non-metallic materials, organic polymer materials, composite materials, or any combination thereof. Conveniently,

there are no limitations on the shape of the initial specimen from which a TEM sample is to be prepared using the present method. For the purpose of TEM analysis, the final sample may be of any shape and size suitable for being held in the TEM sample holder (of the TEM machine). Preferably, the specimen 110 has a bulk thickness of less than 100 nm (nanometers). If the specimen 110 is thicker than 100 nm and/or too big for the TEM sample holder, the specimen 110 may be first cut to a suitable size and/or thinned down using the laser 1040 to a bulk thickness of less than 100 nm, e.g., a thickness in a range from about 1 nm to about 100 nm.

[0018] FIG. 3 and FIGS. 4A-4F are schematic diagrams illustrating embodiments of the method 200 and the various stages of the specimen/sample being formed according to the method 200. According to one embodiment, the method 200 includes ablating (124) using only laser ablation, to form an ablated area (also referred to as a region 1100) in/on the first surface 1182 of a specimen 110.

[0019] Prior to the ablating process 124, the method 200 may involve cutting out a piece of a material from the material or article 109 to be analysed (FIG. 4A). The thinning (122) results in a specimen (FIG. 4B) with a bulk thickness t_0 . Preferably, the thinning process 122 is performed on the first surface 1182 of the specimen 110 exclusively by laser milling using the laser 1040. Preferably, the thinning process 122 is performed exclusively by laser milling over substantially all of the specimen 110 or at least over a substantial portion of the first surface 1182 of the specimen 110.

[0020] For the purpose of the present disclosure, and in comparison to the thinning process 122, the ablating process 124 (FIG. 4C) is relatively localized and subjects a smaller area (referred to as a region 1100) of the specimen 110 to laser processing.

[0021] The thinning process 122 refers to the laser milling that ends with the specimen 110 having a general disk-like or flat shape characterized by a bulk thickness t_0 . In contrast, the ablating process 124 refers to the laser ablation that begins with the specimen 110 being

characterized by the bulk thickness t_0 , and reduces the thickness in one or more regions 1110 of the specimen 110 to a target thickness or a sampling thickness t_s (FIG. 4D or FIG. 4E). It is also possible to produce a foil 300 using the present method 200, in which the ablating (124) results in a specimen 110 with an extended area with the target thickness or sampling thickness t_s (FIG. 4F).

[0022] The specimen may be laser milled at relatively high rate during the thinning (122). The thinning 122 can be configured to a non-thermal process and thus minimizes the damage to the specimen. During the ablating (124), the laser 1040 is configured for relatively low-rate ablation, resulting in little damage to the specimen 110. During the ablating 9124), the laser 1040 is configured as a pulsed laser with pulse duration preferably within a range from 10 femtoseconds to 10 picoseconds. When the ablating (124) is near completion and the remaining material in the region is very thin, the single pulse energy of the pulsed laser is to be set at a significantly low level to reduce the ablation rate.

[0023] In one embodiment, the ablating (124) is performed until it is determined that at least one part of the region 1100 is transparent to electrons. For example, the ablating may continue (126,128) until, in response to the sensor 170 sensing the laser 1040, the controller 102 ends the ablating (126,128). The part of the region that is transparent to electrons is referred to as the penetrated part 128. In some examples, the penetrated part 128 may not include any through holes. In some examples, the penetrated part 128 may include one or more through holes 1076. In some examples, in response to the sensor 170 sensing at least a part of the laser 1040 transmitted through the specimen material or via a through hole 1076, the controller 102 ends the ablating (126,128), or the ablating continues (126,124) in the absence of the sensor 170 sensing the laser 1040. The material at or neighboring the penetrated part 128 (which may be a through hole 1076 or a very thin layer of material that is transparent to electrons) can serve as potential sampling points during TEM analysis. That is, upon the specimen 110 forming at least

one region that has at least one penetrated part 128, the specimen 110 may serve as a TEM sample 150.

[0024] In some embodiments, the laser 1040 may be repositioned to carry out further ablating (130,136,124) to form another ablated region on/in the same first surface 1182 of the same specimen 110. The laser 1040 may be repositioned to a pristine (post laser milling) part of the first surface to form a new ablated region that is non-contiguous with the first region ablated on the same specimen. This may be useful for increasing the likelihood of the TEM analysis providing useful information. For example, in a case where a specimen is suspected of being contaminated, the present method 200 allows TEM to take images at more than one point of the TEM sample 150 and thereby increase the probability of correctly determining whether there is contamination.

[0025] In some embodiments, the laser 1040 may be repositioned to form another laser ablated region that is contiguous with the first ablated region, effectively increasing the area of the ablated region. In the present disclosure, an extended ablated region formed in this manner is referred to as a "foil" 300.

[0026] The present method advantageously provides the user with a choice of being able to perform TEM analysis at one or more sampling points in one sample 150. The present method may further be used to enable formation of a relatively larger area of the target thickness. These are a few examples to illustrate the benefits of the present method 200 in the formation of a TEM sample or a foil (300).

[0027] FIG. 5A is a cross-sectional view of a schematic diagram showing the specimen under laser ablation. FIG. 5B is a magnified partial view of FIG. 5A at an earliest instant when a part of the specimen is transparent to electrons. As illustrated, the actual thickness of the specimen 110 can be non-uniform. As an example, the specimen 110 is shown to have a rough second surface 1184 at microscale or nanoscale. The thickness of the specimen 110 in Fig. 5B

may range from 1 nm to a few hundred nanometers. Oftentimes, even a small degree of heterogeneity in the dimensions and/or material composition can cause the TEM sample to break in unexpected ways in the course of sample preparation. Conventional manual methods of preparing TEM samples therefore require skill, time, and manual effort to slowly grind and polish the TEM sample so as to avoid breaking and destroying the sample. In one sense, in some embodiments, the present method 200 can be said to take a radically unconventional approach by not avoiding breakages in the specimen.

[0028] In the embodiment of FIG. 5A, the specimen 110 is placed on a first substrate 160, and the sensor 170 is provided between the first substrate 160 and a second substrate 162. The first substrate 160 is transparent to the laser 1040. During the ablation process (124), the laser 1040 is configured to follow a helical path, e.g., concurrently following a circular path 1040a and moving along a direction 1040b, along the normal axis 1060 deeper into the specimen 110. At the same time, the laser 1040 is inclined at an inclination angle θ relative to the normal axis 1060 such that a recess 1074 with a slope or inclined surface 1160 is formed. The thickness of the specimen 110 in the region 1100 is reduced at an ablation rate that is configured to be slower than the laser milling rate (in terms of mass of material removed in unit time).

[0029] In some examples, the laser parameters, e.g., laser power, beam spot size, pulse duration, wavelength, etc., are selected within a predetermined range. In some examples, the laser parameters are varied over the ablation process at one region 1100. Preferably, the laser pulse duration is within a range from 10 femtoseconds to 10 picoseconds. Preferably, the laser beam spot size is below 50 microns. Preferably, the laser power is reduced gradually during the ablating 124 as the recess. Preferably, the ablation rate is incrementally decreased over the time duration of the ablating 124 of one region 1100, i.e., the ablation rate decreases over the time taken for ablating one region 1100. In the ablating 124, the laser 1040 may be configured to ablate the specimen 110 along various paths. A few examples are shown in FIGS. 6A to 6C.

For example, the laser 1040 can be configured to sweep the first surface 1182 of the specimen 110 along a circular path 170a (FIG. 6A), a multi-circular path 170b (FIG. 6B), or a spiral path 170c (FIG. 6C). The laser parameters, e.g., power, spot size, and incident angles, may be configured according to the path utilized during the ablation process 124.

[0030] In some examples, the laser 1040 may be configured to be non-transmissive through the material of the specimen 110 unless a through hole is formed in the specimen 110, i.e., the specimen 110 is opaque to the laser 1040. In some examples, the laser 1040 may be transmittable through a sufficiently thin layer of the material of the specimen 110. In FIG. 5B, the laser 1040 is shown as a pulsed laser at one instant when the laser 1040 first penetrates or is at least partially transmissive through the specimen 110, through either one or both of a sufficiently thin layer 1078 of the specimen and a through hole 1076 in the specimen 110. This first penetration of the laser 1040 is sensed by the sensor 170, in response to which the ablating (at this region 1100) is stopped or ends. The system 100 is configured such that immediately upon the sensor 170 first sensing the laser 1040, the laser 1040 is prevented from continuing with the ablation process 124. For example, the controller 102 may instruct the laser source 104 to stop emitting the laser 1040. Alternatively, the specimen 110 may be shielded from the laser 1040.

[0031] The rough second surface 1184 of the specimen 110 which leads to uneven thickness is traditionally considered part of a problem that makes the conventional preparation of a TEM sample technically challenging. Taking an unconventional approach, the irregular and uneven thickness of the specimen 110 becomes part of the solution disclosed herein. The present method 200 gradually decreases the thickness in one region 1100 until some randomly or irregularly distributed parts 128 of the specimen 110 achieve a thickness transmissible by electron beams while other parts 129 are too thick for electron transmission. The region 1100 is formed with at least one very thin part or a through hole (collectively and generally referred

to as a penetrated part 128) while retaining more material at parts neighboring the penetrated part 128. The present method 200 concurrently provides the ablating laser at an inclination angle so that the laser forms a slope between the too thick parts 129 and the sufficiently thin part 128. Stopping the laser 1040 upon at least one penetrated area 128 enables some materials to be left remaining in the region 1100 and these form part of the inclined surface or part of a wedge-shaped material. The present method 200 stops further ablation once a through hole or a sufficient thin layer of material so that small and relatively thin amounts of materials remain in the neighborhood of the penetrated part. The probability of finding sufficiently thin layers of materials is increased nearer each of the at least one penetrated part 128. The inclined laser 1040 facilitates the formation of wedged-shaped material (for the sake of brevity, also referred to as one or more wedges 1088) at the parts neighboring the penetrated part 128. The thinner ends of the wedges 1088 formed in the manner described are likely to provide sufficiently thin material for TEM analysis.

[0032] FIGS. 7A to 7C are cross-sectional views of TEM samples 150 according to embodiments of the present disclosure. FIG. 7B is also a magnified view of a part of FIG. 7A. In the present disclosure, the term "TEM sample" refers to a final form of the specimen 110, i.e., the formed specimen 110 ready for use in TEM analysis.

[0033] The TEM sample 150 of FIG. 7A includes the region 1100 in the form of a recess 1074 in the first surface 1182. The two major surfaces, i.e., the first surface 1182 and the second surface 1184, of the sample 150 define a thickness along the normal axis 1060. During TEM observation, electron beams radiate onto the TEM sample along the thickness direction 1040a. Alternatively, electron beams radiate onto the TEM sample opposite the thickness direction 1040a. The sample 150 has a thickness t_b , which may be referred to as a bulk thickness. The bulk thickness may be in a range from 10 μm to 30 μm , inclusive. The thinning by laser milling to reduce the original thickness t_o of the material to the bulk thickness t_b helps to shorten the

duration of the ablating 124 and keep the probability of failure low. The recess 1074, may be characterized by a non-uniform thickness t_n along an inclined surface 1160 extending from the first surface 1182 to an ablated area 1050 neighboring a penetrated part 128 of the sample 150. The ablated area 1050 may have a thickness that is not uniform, especially at the microscale. The maximum thickness of the ablated area 1050, d_m , is smaller than the bulk thickness, d_b . The recess 116 is produced during ablating 134. The inclined surface 1160 of the recess 116 has a linear profile 1162 in the cross section along the thickness direction 1040a. Although the inclined surface 1160 is shown to be inclined to the two major surfaces 1182, 1184 of the sample 150, the inclined surface 1160 may be perpendicular to the first and second surfaces 1182, 1184 in other examples.

[0034] The ablated area 1050 of the sample 108 have a thickness of less than a sampling thickness d_s , e.g., 100 nm (nanometers) and are transparent for electron beams in TEM characterization. The term “sampling thickness” refers to a thickness below which electron beams are transmissive through a sample and above which electron beams are not transmissive through the sample. In other words, a sample with a thickness above a sampling thickness is not visible under TEM observation while a sample with a thickness below a sampling thickness is visible. Each of the wedge-shaped materials 1088 near a through hole 1076 provides at least one ablated area 1050 at a thin end 1090 of the respective wedge-shaped materials 1088. Although FIG. 7B shows a through hole 1076 in the sample 150, the sample 150 does not necessarily need a through hole. The TEM sample 150 of FIG. 7C does not have a through hole in the ablated area 1050 (at the thinnest part of the recess 1074) but it can be used for TEM analysis because there are areas thinner than the sampling thickness t_s . One or more sampling points 1092 for the TEM analysis may be selected from the ablated area 1050.

[0035] As the laser beam of the system 100 is rotated instead of the specimen, recesses can be created at different locations by moving the laser head. FIG. 8 illustrates a sectional

perspective view of a TEM sample 118 according to one embodiment of the present disclosure. The TEM sample 118 can have one or more regions 110 or recesses 1074 at one surface 1182 of the specimen. The TEM sample 118 has two major surfaces 1182 and 1184. During TEM observation, electron beams may radiate onto the TEM sample 118 along the direction 118a. The TEM sample 118 has a disk shape with a diameter S_0 and a bulk thickness, t_b . Multiple recesses 180, 182, 184, 186, and 188 may be formed in the TEM sample 118 at/in the first surface 1182. Using such a TEM sample, the likelihood of successfully conducting the TEM analysis and obtaining useful results increases. The multiple regions 1100 may be configured differently from one to another.

[0036] The recess 180 has a cylindrical profile with a diameter, D_1 , while the recess 182 also has a cylindrical sectional profile with a diameter, D_2 . D_2 is smaller than D_1 . The recess 184 has a trapezoidal sectional profile with a tapered angle, α_1 , while the recess 186 has a trapezoidal sectional profile with a tapered angle, α_2 , in which α_1 is larger than α_2 . The recess 188 has a reversed trapezoidal sectional profile, i.e., a shorter side of the reversed trapezoidal sectional profile is formed on the first surface 1182 while a longer side is formed near the second surface 1184. The recess 188 is formed by ablating 144 with a laser inclined to enter the opening of the recess 188 on the surface 1182. The recesses 184, 186, and 188 with tapered angles broaden transparent regions of the TEM sample 118 for electron beam and so enables TEM users to quickly find out the target regions, especially at low magnification mode. It should be understandable that although each of the recesses 180, 182, 184, 186, 188 may have a symmetric geometry or each of them may have an asymmetric geometry.

[0037] A conventional TEM bulk sample fabricated with conventional approaches have only one ablated area, which may not be transparent for electron beams. In contrast, the TEM sample 118 as disclosed herein provides multiple ablated regions and thus increases the probability of

success in TEM observation. In other words, the multiple ablated regions provided by the TEM sample 118 increases the probability of electron beam transmission.

[0038] As described above, the method 200 may include providing the laser 1040 at an angular displacement 1044 relative to a normal axis 1060 after the thinning 122 and before the ablating 124 such that the laser 1040 is directed at the specimen 110 at an inclination angle θ during the ablating 126, wherein the laser 1040 is directed at the specimen 110 along the normal axis 1060 during the thinning 122. The method 200 may further include selecting a selected plurality of the region 1100 in the first surface 1182, and repeating the ablating 124 and the ending 128 thereof for each of the selected plurality of the region 1100. The method 200 may further include reconfiguring the inclination angle θ for the ablating 124 of different ones of the selected plurality of the region 1100, such that the different ones of the selected plurality of the region are each characterized by differently inclined surfaces. The ablating 124 forms a recess 116 in the region 1100 in the first surface 152. The recess 116 is at least partially defined by an inclined surface 1160 with a linear profile 1162 corresponding to one of the paths 170a, 170b, or 170c of the laser 1040 performing the ablating 134. The recess 116 is characterized by a non-uniform thickness, t_n , ranging from the bulk thickness t_b to a sampling thickness t_s , the sampling thickness being a thickness at which the specimen is transparent to the electrons. The sampling thickness, t_s , is in a range from 30 nanometers (nm) to 50 nm, inclusive. The recess 116 includes one or more wedge-shaped material 1088 providing at least one ablated area 1050 at a thin end 1090 of the one or more wedge-shaped material 1088. The at least one penetrated part 128 may be a through hole 1076. The ablated area 1050 neighboring the through hole 1076 is characterized by the sampling thickness, d_s . The method 200 may further comprise providing a sensor, e.g., the photosensor 170, to sense only a transmitted part 1042 of the laser 1040, and ending the ablating upon the sensor first sensing the transmitted part 1042 of the laser 1040 during the ablating. The method 200 may further include before the ablating 134, cutting the

specimen from a bulk material such that the specimen is accommodatable in a TEM sample holder of the TEM. The bulk thickness t_b is in a range from 10 micrometers (μm) to 30 μm , inclusive. For the ablating 124, the laser 1040 may be configured to follow a helical scanning path 170c. For the ablating 124, the laser 1040 may be configured as an ultrashort pulsed laser. The laser 1040 may be further configured with a single pulse energy that decreases over the ablating 134 of the region 1100.

[0039] A TEM sample for transmission electron microscope (TEM) analysis prepared according to the present method 200 includes one or more regions 1100 forming a respective recess 1074 in a first surface 1182, the respective recess 1100 being characterized by a non-uniform thickness t_n along an inclined surface 1160 extending from the first surface to an ablated area neighboring a penetrated part 128, in which the penetrated part 128 is characterized by a sampling thickness that is transparent to electrons.

[0040] The present disclosure describes a foil 300 prepared according to the method 200. The foil 300 includes a penetrated part 128, in which the penetrated part is characterized by a sampling thickness that is transparent to electrons. The sampling thickness may be in a range from 30 nanometers (nm) to 50 nm, inclusive (i.e., inclusive of 30 nm as well as 50 nm). The at least one penetrated part may include or may define a through hole 1076.

[0041] All examples described herein, whether of apparatus, methods, materials, or products, are presented for the purpose of illustration and to aid understanding, and are not intended to be limiting or exhaustive. Various changes and modifications may be made by one of ordinary skill in the art without departing from the scope of the invention as claimed.

Claims

1. A method of preparing a sample for transmission electron microscope (TEM) analysis, the method comprising:
 - thinning a specimen to a bulk thickness that is non-transparent to electrons, the thinning being performed on a first surface of the specimen exclusively by laser milling using a laser;
 - ablating a region in the first surface of the specimen such that the region is thinner than the bulk thickness, the ablating being performed exclusively by the laser within the region to leave other parts of the first surface unmodified by the ablating; and
 - ending the ablating in response to sensing that at least one penetrated part of the region is transparent to electrons, such that a sampling point for the TEM analysis is selectable from an ablated area at or neighboring the at least one penetrated part.
2. The method according to claim 1, further comprising: providing the laser at an angular displacement relative to a normal axis after the thinning and before the ablating such that the laser is directed at the specimen at an inclination angle during the ablating, wherein the laser is directed at the specimen along the normal axis during the thinning.
3. The method according to claim 2, further comprising:
 - selecting a selected plurality of the region in the first surface; and
 - repeating the ablating and the ending thereof for each of the selected plurality of the region.
4. The method according to claim 3, further comprising: reconfiguring the inclination angle for the ablating of different ones of the selected plurality of the region, such that the different ones of the selected plurality of the region are each characterised by differently inclined surfaces.
5. The method according to claim 1 or claim 2, wherein the ablating forms a recess in the region in the first surface.

6. The method according to claim 5, wherein the recess is at least partially defined by an inclined surface with a linear profile corresponding to a path of the laser performing the ablating.
7. The method according to claim 5 or claim 6, wherein the recess is characterized by a non-uniform thickness ranging from the bulk thickness to a sampling thickness, the sampling thickness being a thickness at which the specimen is transparent to the electrons.
8. The method according to claim 7, wherein the sampling thickness is in a range from 30 nm to 50 nm, inclusive.
9. The method according to any one of claims 5 to 8, wherein the recess comprises one or more wedge-shaped material providing at least one ablated area at a thin end of the one or more wedge-shaped material.
10. The method according to any one of claims 1 to 9, wherein the at least one penetrated part comprises a through hole.
11. The method according to claim 10, wherein the ablated area neighboring the through hole is characterised by the sampling thickness.
12. The method according to any one of claims 1 to 11, comprising:
 - providing a sensor to sense only a transmitted part of the laser; and
 - ending the ablating upon the sensor first sensing the transmitted part of the laser during the ablating.
13. The method according to any one of claims 1 to 12, comprising:
 - before the ablating, cutting the specimen from a bulk material such that the specimen is accommodatable in a TEM sample holder of the TEM.

14. The method according to any one of the claims 1 to 13, wherein the bulk thickness is in a range from 10 μm to 30 μm , inclusive.
15. The method according to any one of claims 1 to 14, wherein for the ablating, the laser is configured to follow a helical scanning path in the ablating.
16. The method according to any one of claims 1 to 15, wherein for the ablating, the laser is configured as an ultrashort pulse laser.
17. The method according to claim 16, wherein the laser is configured with a single pulse energy that decreases over the ablating of the region.
18. A sample for transmission electron microscope analysis prepared by the method of any one of the claims 1 to 17, the sample comprising:
one or more regions each forming a respective recess in a first surface, the respective recess being characterized by a non-uniform thickness along an inclined surface extending from the first surface to an ablated area neighboring a penetrated part, wherein the penetrated part is characterized by a sampling thickness that is transparent to electrons.
19. A method of preparing a foil of a material, the method comprising:
thinning a specimen to a bulk thickness, the thinning being performed on a first surface of the specimen exclusively by laser milling using a laser;
ablating a first region in the first surface of the specimen such that the first region is thinner than the bulk thickness, the ablating being performed exclusively by the laser;
ending the ablating in response to sensing that at least one penetrated part of the first region is at or thinner than a target thickness at which the material is transparent to electrons; and
repeating the ablating and the ending thereof at another region contiguous with the first region until a predetermined contiguous area of the first surface is transparent to electrons.

20. A foil formed according to the method of claim 19, wherein the foil comprises a predetermined contiguous area having a foil thickness that is transparent to electrons.
21. The foil according to claim 20, wherein the foil thickness is 50 nm or thinner than 50 nm.