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(54) Title: SAMPLE HOLDER FOR ELECTRON MICROSCOPY FOR LOW-CURRENT, LOW-NOISE ANALYSIS

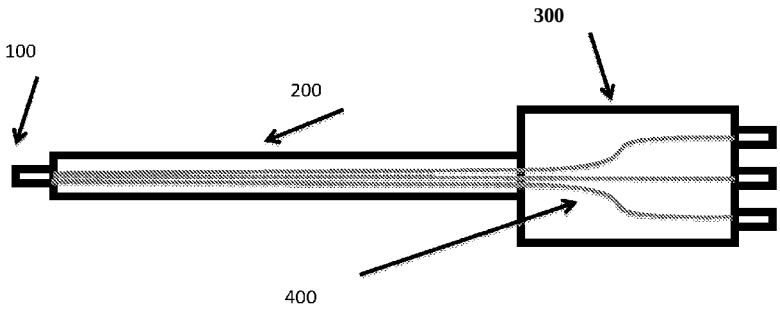

FIGURE 2

(57) Abstract: A novel specimen holder for insertion in electron microscopes, wherein the novel specimen holder is designed to minimize electrical noise so that signal integrity can be maintained during in situ electron microscopy.

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SAMPLE HOLDER FOR ELECTRON MICROSCOPY FOR LOW-CURRENT, LOW-NOISE ANALYSIS

Field

[0001] The invention relates generally to specimen holders used for mounting samples in an electron microscope, e.g., a transmission electron microscope (TEM), a scanning transmission electron microscopy (STEM) and variations of the scanning electron microscopes (SEM) that use traditional TEM-type holders and stages, for low-current and low-noise imaging and analysis.

Background

[0002] The specimen holder is a component of an electron microscope providing the physical support for specimens under observation. Specimen holders traditionally used for TEMs and STEMs, as well as some modern SEMs, consist of a rod that is comprised of three key regions: the end (300), the barrel (200) and the specimen tip (100) (see, e.g., Figure 1). In addition to supporting the specimen, the specimen holder provides an interface between the inside of the instrument (i.e., a vacuum environment) and the outside world.

[0003] To use the specimen holder, one or more samples are first placed on a specimen support device. The specimen support device is then mechanically fixed in place at the specimen tip, and the specimen holder is inserted into the electron microscope through a load-lock. During insertion, the specimen holder is pushed into the electron microscope until it stops, which results in the specimen tip of the specimen holder being located in the column of the microscope. At this point, the barrel of the specimen holder bridges the space between the inside of the microscope and the outside of the load lock, and the end of the specimen holder is outside the microscope. To maintain an ultra-high vacuum environment inside the electron microscope, flexible o-rings are typically found along the barrel of the specimen holder, and these o-rings seal against the microscope when the specimen holder is inserted. The exact shape and size of the specimen holder varies with the type and manufacturer of the electron microscope, but each holder contains these three key regions.

[0004] The specimen holder can also be used to provide stimulus to the specimen, and this stimulus can include temperature (heating or cooling), electrical (e.g., applying a voltage or current), mechanical (e.g., applying stress or strain), chemical (e.g., containing a sample in a specific chemical environment), gas (e.g. containing a sample in a specific gaseous environment) or several at once. For example, an electrical power supply can generate a current or voltage waveform, while a syringe pump can be used hold and force liquids. This equipment is outside of the microscope, and various connectors are used to bring this stimulus to the holder, down the length of the holder, and to the samples. For example, microfiuidic tubing can be used to supply liquids from the syringe pump to the

sample, while electrical wires can be used to supply voltage/current. The specimen holder can also be used to deliver electrical signals measured from a sensor near the sample or the sample itself (e.g., electrical currents and/or voltages).

[0005] It is an object of the present invention to introduce an electron microscopy holder especially designed for in situ electrical stimuli and/or measurements, wherein said holder is improved to minimize electrical noise so that signal integrity can be maintained.

Summary

[0006] The present invention relates generally to a novel specimen holder which minimizes electrical noise so that signal integrity can be maintained, especially when small signals are provided to a sample or measured from a sample.

[0007] Other aspects, features and embodiments of the invention will be more fully apparent from the ensuing disclosure and appended claims.

Brief Description of Figures

[0008] Figure 1 shows a schematic of a generic specimen holder. The specimen holder is comprised of three regions: the tip (100), the barrel (200), and the end (300).

[0009] Figure 2 shows an embodiment of the apparatus described herein, wherein the apparatus comprises shielded cables.

[0010] Figure 3 shows an embodiment of the apparatus described herein, wherein the apparatus comprises shielded cables and an amplifier.

[0011] Figure 4 shows an embodiment of the apparatus described herein, wherein the apparatus comprises shielded cables and a A/D signal converter.

Detailed Description

[0012] The present invention generally relates to novel electron microscopy specimen holders, especially for use during in situ electron microscopy where an electrical stimulus is applied. It is to be understood that the specimen holder described herein is compatible with and may be interfaced with the specimen support devices, e.g., semiconductor specimen support devices, disclosed in International Patent Application Nos. PCT/US08/63200 filed on May 9, 2008 and PCT/US08/88052 filed on December 22, 2008, which are both incorporated herein by reference in their entireties. For example, the semiconductor specimen support devices can be membrane based MEMS devices. Further, it is understood that the specimen holder described herein may include the novel specimen holder tips disclosed in U.S. Patent Application No. 13/085,273 filed on April 12, 2011 and

PCT/USl 1/46282 filed on August 2, 201 1, which are also incorporated herein by reference in their entireties. The specimen holder can be manufactured with tips, barrels and ends of various shapes and sizes such that the specimen holder fits any manufacturer's electron microscope.

[0013] The general area of "in situ" electron microscopy involves applying stimulus to a sample during imaging. The stimulus could be thermal (heating or cooling), electrical (applying a voltage or current), mechanical (applying stress or strain), chemical (containing a sample in a specific chemical environment), or several of these at once.

[0014] Samples are introduced into the microscope using a "holder." The holder places the sample into the microscope at the correct position for imaging. For in situ electron microscopy, the holder is also used to provide an interface between the sample and equipment located outside the microscope that is used to provide the stimulus. For example, an electrical power supply can generate a current or voltage waveform. This equipment is outside of the microscope, and various connectors are used to bring this stimulus to the holder, down the length of the holder, and to the samples. For example, electrical wires can be used to supply voltage/current.

[0015] When samples require very small electrical signals for either stimuli or measurement, for example millivolt (or lower) voltages and/or nanoamp (or lower) currents, small signals have to be provided to the sample and the response measured. Disadvantageous^, noise can present due to electromagnetic interference from other conductors in the holder or from sources external to the holder, e.g., other equipment in the microscopy laboratory. Noise can also be coupled into the conductors from power sources near the holder. That said, electrical noise should be minimized so that signal integrity can be maintained so that the electrical response is measurable.

[0016] In one aspect, an apparatus for minimizing electrical noise at an electron microscopy holder is described, said apparatus comprising an electron microscopy specimen tip and at least one of a low-noise power supply, at least one shielded cable, at least one triaxial/coaxial connector, an amplifier/pre-amplifier, an A/D and/or D/A converter, or any combination thereof.

[0017] With regards to the low-noise power supply, power supplies are available with different minimum voltage/current specifications. For low current/low noise measurements, the lowest possible ratings for these parameters should be chosen when selecting a power supply. For example, a current rating below about 1 μA is preferred.

[0018] With regards to the shielded cabling, cables that typically run from the power supply to the connectors on the holder handle, down the length of the holder rod, to the specimen tip, can be shielded, e.g., a coaxial or triaxial cable that has a center conductor and a layer (or layers) of shielding surrounding the center conductor. As described above, shielded cables protect the signal lines from electrical noise. A shielded cable can either reflect the noise or conduct it to ground, depending on how the shield is connected. Cables are typically shielded with metal foil or braided metal wires. The metal foils or wires are then covered with an insulating plastic sheath. In a preferred embodiment, one shielded cable would be required for each electrical contact in the holder tip.

[0019] With regards to the triaxial/coaxial connectors, preferably high quality triaxial/coaxial connectors are used to connect all electrical cabling. Connectors can include BNC connectors, F connectors, N connectors, and SMA connectors.

[0020] With regards to the amplifier/preamplifier, a high-quality amplifier/preamplifier located in the TEM holder handle can amplify the signal to increase its signal-to-noise ratio and make it less susceptible to noise during transmission from the holder prior to measurement. "Low-noise" amplifiers that provide large gains with minimal noise are ideal. For example, the National Instruments LMC6001Ultra-low input current amplifier (or similar products) can be used.

[0021] With regards to analog-to-digital (A/D) and/or digital-to-analog (D/A) converters, A/D converters can be used to digitize the output signal from the sample and make it less susceptible to noise during transmission from the holder. These converters can also be located in the TEM holder handle. For example, the Texas Instruments DDC112 Dual Current Input 20-bit A/D Converter can be used. For very small signals, an amplifier (described above) can be used to amplify the signal before it enters the A/D converter. In this case, both the amplifier and the A/D converter can be located in the TEM holder handle.

[0022] In one embodiment, the electron microscopy holder comprises the specimen tip (100), and a set of shielded cables (400), as shown schematically in Figure 2. The shielded cables carrying electrical signals from the holder handle to the specimen tip minimize noise/interference from other signal lines and/or the outside environment. Preferably, the holder further comprises a low-noise power supply and triaxial/coaxial connectors. Accordingly, in a preferred embodiment, the electron microscopy holder comprises the specimen tip (100), a low-noise power supply, triaxial/coaxial connectors, and a set of shielded cables (400). When in use, the electron microscopy holder accommodates a specimen device, wherein the specimen device is interfaced with the specimen tip. Preferably the specimen device comprises a membrane-based MEMS device.

[0023] In another embodiment, the electron microscopy holder comprises the specimen tip (100), a set of shielded cables (400) and an amplifier (500) in the holder handle (300), as shown schematically in Figure 3. The amplifier preferably increases the magnitude of small electrical signals. Preferably, the holder further comprises a low-noise power supply and triaxial/coaxial connectors. Accordingly, in a preferred embodiment, the electron microscopy holder comprises the specimen tip (100), a low-noise power supply, a set of shielded cables (400), triaxial/coaxial connectors, and an amplifier (500) in the holder handle (300). When in use, the electron microscopy holder accommodates a specimen device, wherein the specimen device is interfaced with the specimen tip. Preferably the specimen device comprises a membrane-based MEMS device.

[0024] In still another embodiment, the electron microscopy holder comprises the specimen tip (100), a set of shielded cables (400) and an analog-to-digital signal converter (600) in the handle (300), as shown schematically in Figure 4. The A/D converter preferably provides digital output that can then be transmitted with less susceptibility to noise. Preferably, the holder further comprises a low-noise

power supply and triaxial/coaxial connectors. Accordingly, in a preferred embodiment, the electron microscopy holder comprises the specimen tip (100), a low-noise power supply, triaxial/coaxial connectors, a set of shielded cables (400) and an analog-to-digital signal converter (600) in the handle (300). When in use, the electron microscopy holder accommodates a specimen device, wherein the specimen device is interfaced with the specimen tip. Preferably the specimen device comprises a membrane-based MEMS device.

[0025] In yet another embodiment, the electron microscopy holder comprises the specimen tip, a set of shielded cables, an amplifier and an analog-to-digital signal converter in the handle. The amplifier preferably increases the magnitude of small electrical signals and the A/D converter preferably provides digital output that can then be transmitted with less susceptibility to noise. Preferably, the holder further comprises a low-noise power supply and triaxial/coaxial connectors. Accordingly, in a preferred embodiment, the electron microscopy holder comprises the specimen tip, a low-noise power supply, triaxial/coaxial connectors, a set of shielded cables, an amplifier and an analog-to-digital signal converter in the handle. When in use, the electron microscopy holder accommodates a specimen device, wherein the specimen device is interfaced with the specimen tip. Preferably the specimen device comprises a membrane-based MEMS device.

[0026] It should be appreciated that the electron microscopy holders described herein can be adapted for use in liquid, gas or vacuum environments, as readily understood by the person skilled in the art.

[0027] In another aspect, a method of minimizing electrical noise in an electron microscopy holder is described, said method incorporating at least one electrical noise minimizer in said electron microscopy holder, wherein said electrical noise minimizer is selected from the group consisting of a low-noise power supply, at least one shielded cable, at least one triaxial/coaxial connector, an amplifier/pre-amplifier, an A/D and/or D/A converter, and any combination thereof.

[0028] Although the invention has been variously disclosed herein with reference to illustrative embodiments and features, it will be appreciated that the embodiments and features described hereinabove are not intended to limit the invention, and that other variations, modifications and other embodiments will suggest themselves to those of ordinary skill in the art, based on the disclosure herein. The invention therefore is to be broadly construed, as encompassing all such variations, modifications and alternative embodiments within the spirit and scope of the claims hereafter set forth.

What is claimed is:

1. An electron microscope specimen holder comprising a specimen tip and at least one electrical noise minimizer, wherein said electrical noise minimizer is selected from the group consisting of a low-noise power supply, at least one shielded cable, at least one triaxial/coaxial connector, an amplifier/pre-amplifier, an A/D and/or D/A converter, and any combination thereof.

2. The electron microscope specimen holder of claim 1, comprising the low-noise power supply, at least one triaxial/coaxial connector, and at least one shielded cable.

3. The electron microscope specimen holder of claims 1 or 2, wherein the at least one shielded cable comprises internal wiring extending from an external connector to a region in proximity of the specimen tip.

4. The electron microscope specimen holder of any of the preceding claims, further comprising an amplifier/preamplifier, an A/D and/or D/A converter, or both the amplifier/preamplifier and an A/D and/or D/A converter.

5. The electron microscope specimen holder of any of the preceding claims, wherein a specimen device can interface with the specimen tip.

6. A method of minimizing electrical noise in an electron microscopy holder, said method comprising incorporating at least one electrical noise minimizer in said electron microscopy holder, wherein said electrical noise minimizer is selected from the group consisting of a low-noise power supply, at least one shielded cable, at least one triaxial/coaxial connector, an amplifier/pre-amplifier, an A/D and/or D/A converter, and any combination thereof.

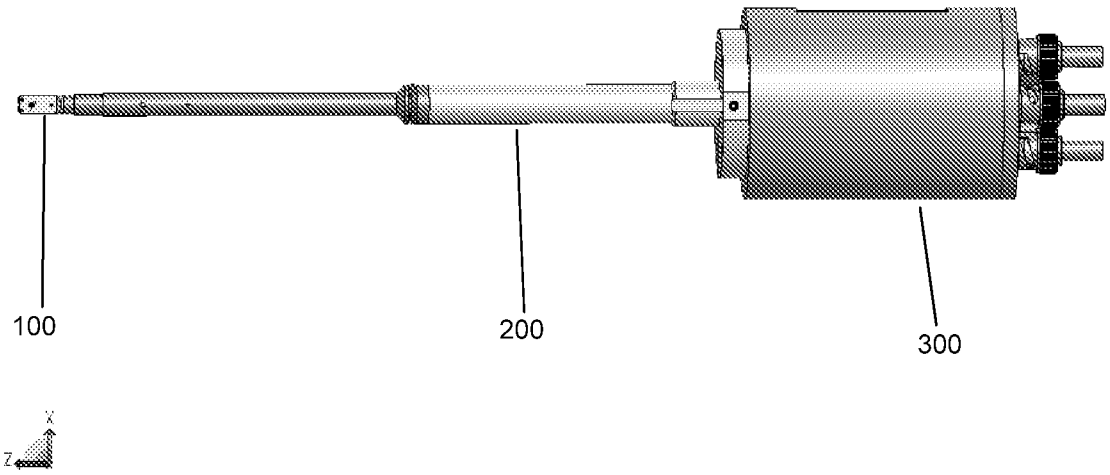

FIGURE 1

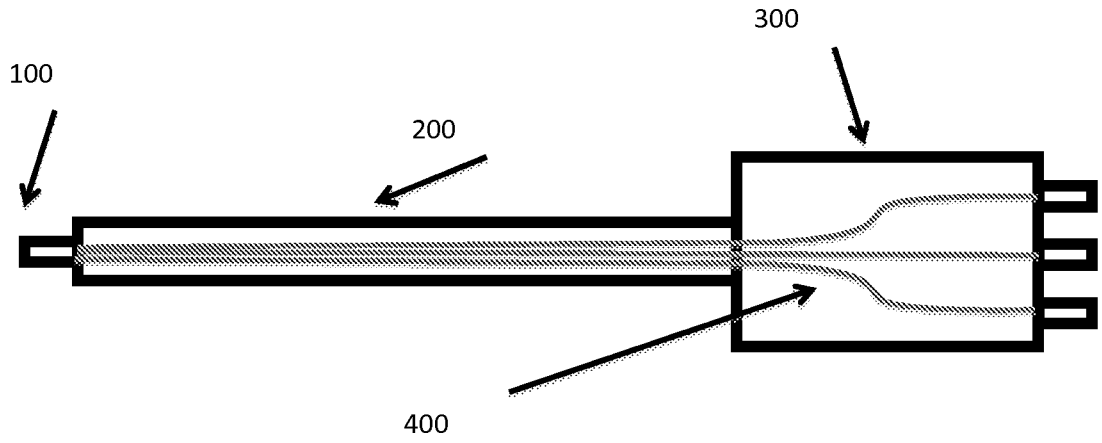

FIGURE 2

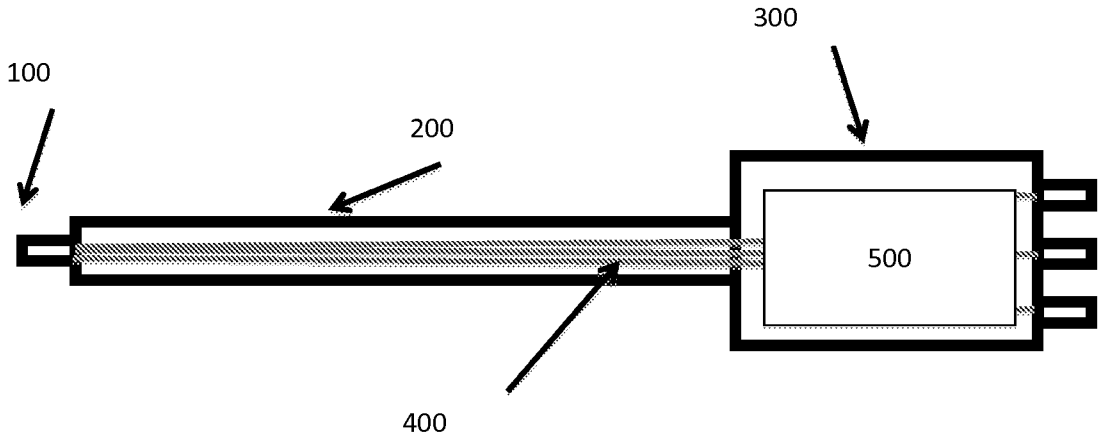

FIGURE 3

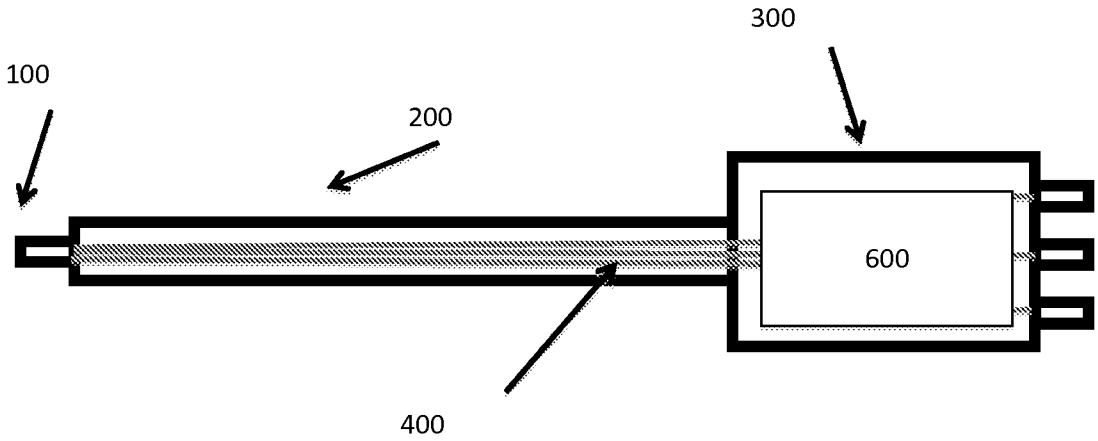

FIGURE 4

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2012/072050

A. CLASSIFICATION OF SUBJECT MATTER

HOIJ 37/20(2006.01)i, H01B 11/06(2006.01)1

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
HOIJ 37/20; G01N 23/225; G21K 5/08; HOIJ 37/244; G01L 5/16; HOIJ 3/14; HOIJ 37/28

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
Korean utility models and applications for utility models
Japanese utility models and applications for utility models

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
eKOMPASS(KIPO internal) & Keywords: microscope, sample holder, converter, amplifier, shielded cable, noise, and similar terms.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 2002-0008201 Al (YUKIHIRO TANAKA et al.) 24 January 2002 See paragraphs [0005], [0007], [0031], and [0043]-[0045]; claims 1-2; and figures 1-5.	1-3,6
Y	US 2011-0248165 Al (JOHN DAMIANO, JR. et al.) 13 October 2011 See paragraphs [0005], [0025] and [0032]-[0033]; claims 1, 4, and 8; and figures 1-3.	1-3,6
A	US 2004-0061066 Al (KEN HARADA et al.) 1 April 2004 See paragraphs [0054] and [0140]-[0141]; claim 1; and figure 7.	1-3,6
A	US 2010-0090109 Al (MICHIO HATANO et al.) 15 April 2010 See paragraphs [0028]-[0029]; and figure 1.	1-3,6
A	WO 2010-112242 Al (FEMTOTOOLS GMBH et al.) 7 October 2010 See paragraphs [0014], [0022], and [0052]; and figures 1-4.	1-3,6

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier application or patent but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
29 April 2013 (29.04.2013)	**29 April 2013 (29.04.2013)**

Name and mailing address of the ISA/KR
Korean Intellectual Property Office
189 Cheongsa-ro, Seo-gu, Daejeon Metropolitan City, 302-70 1, Republic of Korea
Facsimile No. 82-42-472-7140

Authorized officer
NHO, Ji Myong
Telephone No. 82-42-481-8528

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2012/072050

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☒ Claims Nos.: 4-5
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box No. Ill Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required addtional search fees were timely paid by the applicant, this international search report covers all searchable claims.

2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

☐ The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.

☐ The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.

☐ No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/US2012/072050

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