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(54) OPTICAL SENSOR PLATFORM EMPLOYING HYPERBOLIC METAMATERIALS

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(2013.01); B01L 3/502761 (2013.01); (Continued)
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(57) ABSTRACT

Disclosed herein are optical sensor platform(s) employing hyperbolic metamaterial(s) supporting highly confined bulk plasmon guided modes over broad wavelength range(s) from visible to near-infrared. By exciting these modes using—for example - a two-dimensional $(2D)$ grating coupling technique, sensors according to the present disclosure advantageously exhibit extreme sensitivity modes up to a maximum of 30,000 nm per refractive index unit and a record figure of (2014.01) merit of 590 thereby permitting detection of ultralow-mo-
 GUN 21/552 (2014.01) lecular-weight bio-molecules at picomolar concentrations.

(Continued) 9 Claims, 17 Drawing Sheets

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- CPC **G01N 21/553** (2013.01); **G01N 21/7743** $(2013.01);$ GOIN 33/54346 (2013.01); GOIN 33/54373 (2013.01); B01L 2300/0654 (2013.01); B01L 2300/0887 (2013.01)
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FIG. 1

FIG. 2

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FIG .3(G)

Sheet 13 of 17

Patent Application Ser. No. 62/378,464 filed 23 Aug. 2016 random distribution of national properties according to a spectrum of $\frac{10}{\text{m}}$ for a length $\frac{10}{\text{m}}$ for a length $\frac{10}{\text{m}}$ for a length $\frac{10}{\text{m}}$ which is incorporated by reference as if set forth at length 10 the present disclosure;
FIGS. 4(A) and 4(B) schematically show a top-view and
herein.

This disclosure relates generally to sensor technologies 15 aspect of the present disclosure;
d more particularly to an optical sensor platform employ. FIG. 5 is a photograph showing the GC-HMM sensor and more particularly to an optical sensor platform employ-
ing hyperbolic metamaterials.
device of FIG. 2 with microfluidic channel and sample

As will be readily appreciated by those skilled in the art, bolic dispersion at tical sensor technologies offer significant opportunities in present disclosure; optical sensor technologies offer significant opportunities in present disclosure;
the chemical, environmental, biological and medical FIG. 7 is a plot showing reflectance spectra of the the chemical, environmental, biological and medical FIG 7 is a plot showing reflectance spectra of the research and diagnostic field(s)—among others—particu- GC-HMM at different angles of incidence according to an research and diagnostic field(s)—among others—particu-
larly at different angles of incidence according to an analysis of 25 aspect of the present disclosure; and larly with respect to the detection of small numbers of 25 aspect of the present disclosure; and molecules in highly dilute solutions. Given their potential FIG. 8 is a schematic block diagram illustrating the importance, new and/or improved optical sensor technolo-
gies would represent a welcome addition to the art.
disclosure into higher order instrumentation systems.

the present disclosure directed to an optical sensor platform disclosure. It will thus be appreciated that those skilled in the employing hyperbolic metamaterials. In sharp contrast to art will be able to devise various ar contemporary optical sensor technologies and sensors con- 35 although not explicitly described or shown herein, embody
structed therefrom, optical sensors according to the present the principles of the disclosure and are i highly confined bulk plasmon guided modes over broad details are set forth, it is understood that embodiments of the wavelength range(s) from visible to near-infrared. By excit-
disclosure may be practiced without these sp wavelength range(s) from visible to near-infrared. By excit-
ing these modes using—for example—a two-dimensional 40 and in other instances, well-known circuits, structures and ing these modes using—for example—a two-dimensional 40 and in other instances, well-known circuits, structures and (2D) grating-coupling technique, a metalized methyl meth-
(2D) grating-coupling technique, a metalized meth (2D) grating-coupling technique, a metalized methyl meth-
acrylate structure, and/or a random distribution of nanopar-
understanding of this disclosure.

A more complete understanding of the present disclosure recited examples and conditions.

may be realized by reference to the accompanying drawing 50 Moreover, all statements herein reciting principles,

in which:

FIG. 1

bolic metamaterial sensor (HMM) structure according to an structural and functional equivalents thereof. Additionally, it aspect of the present disclosure; it is intended that such equivalents include both currently-

fluid flow channel according to an aspect of the present function, regardless of structure.
disclosure;
FIGS. 3(A)-3(C) show schematics of alternative HMM in the art that the diagrams herein represent conceptual

sensor device(s) that are fabricated in a lithography-free 60 views of illustrative structures embodying the principles of manner wherein $3(A)$ employs a hybrid partially leaky the disclosure. waveguide (HPLW) overlying the HMM stack; 3(B) In the claims hereof any element expressed as a means for employs a submicron layer of methyl methacrylate coated performing a specified function is intended to encompass with a thin (5-15 nm) layer of metal (Au or Pd); and $3(C)$ any way of performing that function including, for example, employs a random distribution of nanoparticles—i.e., 100 65 a) a combination of circuit elements which present disclosure; https://www.more.more aspects of the function or b in any form α in any form α

OPTICAL SENSOR PLATFORM FIG. 3(D) shows (left) a theoretically calculated disper-
 EMPLOYING HYPERBOLIC sion diagram for transverse magnetic modes (dotted lines) in

a lithography-free HMM system in terms of photon energ a lithography-free HMM system in terms of photon energy versus momentum and (right) electric field magnitude for CROSS REFERENCE TO RELATED $\frac{5 \text{ three modes located at A, B, C in left plot—according to} {\text{appulCATIONS}}$ aspects of the present disclosure;

FIGS. $3(E)$ - $3(J)$ show intensity vs wavelength graphs and corresponding magnetic field plots for structures including This application claims the benefit of U.S. Provisional corresponding magnetic field plots for structures including
tent Application Ser No. 62/378.464 filed 23 Aug 2016 random distribution of nanoparticles according to as

> side-view respectively, of an illustrative alternative embodi-TECHNICAL FIELD ment of an HMM sensor device with integrated microfluidic reservoir, channels and filter structures according to an aspect of the present disclosure;

ing according to an aspect of the present disclosure;
BACKGROUND FIG. 6 is a plot showing permittivity of gold/Al₂O₃HN

FIG. 6 is a plot showing permittivity of gold/ Al_2O_3 HMM
20 determined using effective media theory showing a hyperdetermined using effective media theory showing a hyper-
bolic dispersion at 520 nm according to an aspect of the

SUMMARY 30 DETAILED DESCRIPTION

An advance in the art is made according to an aspect of The following merely illustrates the principles of the present disclosure directed to an optical sensor platform disclosure. It will thus be appreciated that those sk

acrylate structure, and/or a random distribution of hanopar-
ticles, sensors according to the present disclosure advanta-
geously detect ultralow-molecular-weight bio-molecules at accided herein are principally intended ex BRIEF DESCRIPTION OF THE DRAWING by the inventor(s) to furthering the art, and are to be construed as being without limitation to such specifically

FIG. 1 shows a schematic of a generalized optical, hyper-
bolic metamaterial sensor (HMM) structure according to an structural and functional equivalents thereof. Additionally, it FIG. 2 shows a schematic of a 2D grating-coupled hyper- 55 known equivalents as well as equivalents developed in the bolic metamaterial (GC-HMM) sensor device including a future, i.e., any elements developed that perform t

FIGS. 3(A)-3(C) show schematics of alternative HMM in the art that the diagrams herein represent conceptual sensor device(s) that are fabricated in a lithography-free 60 views of illustrative structures embodying the princ

which the claims call for. Applicant thus regards any means 5 that the functionalities provided by the various recited and hole sizes can be designed and fabricated to match the means are combined and brought together in the manner relative optical momenta.

in the art that the diagrams herein represent conceptual 10 readily appreciate that liquids and/or gases may be delivered
views of illustrative structures embodying the principles of
the disclosure.

FIG. 1 shows an illustrative generalized schematic of an to the present disclosure.

optical hyperbolic metamaterial (HMM) sensor structure Advantageously, and as will be readily appreciated by

according to aspects of the according to aspects of the present disclosure. As shown in 15 those skilled in the art, a GC-HMM sensor structure according to the present disclosure may be fabricated using well prising a number of bilayers (layer-pairs)—overlies a sub-
strate. Overlying the HMM stack is a dielectric layer over-
of illustrative example using the structure depicted in FIG. lying which is an optical "coupler" structure. An analyte 2, a GC-HMM sensor structure according to the present
delivery structure overlies the coupler structure such that 20 disclosure may be fabricated by sequential dep may be optically detected as a result of its effect upon films by using conventional electron beam and thermal surface plasmon/bulk plasmon interactions occurring within evaporation techniques, respectively. All the thin f surface plasmon/bulk plasmon interactions occurring within evaporation techniques, respectively. All the thin films may
the HMM stack.
All the thin films may be grown over micro-glass substrates with Al₂O₂ and Au

illustrative, fabricated, miniaturized GC-HMM sensor Operationally, a solution including solute(s) to be device according to an aspect of the present disclosure. As detected enters an input of the microfluidic channel, flo may be observed from that FIG. 2, the illustrative GC-HMM across/within the GC-HMM, and subsequently exits an sensor device includes a microfluidic flow channel overlying output. Due to its structure, the microfluidic chan sensor device includes a microfluidic flow channel overlying a grating coupled metamaterial.

Illustratively, the metamaterial shown includes a stack of GC-HMM sensor structure. As a result of this contact, a eight (8) Au/Al₂O₃ layer-pairs. Overlying the eight layer-
number of the solute molecules are adsorb pair stack is a dielectric spacer layer which in turn is overlaid surface of the sensor structure. Advantageously, and with with a Gold, 2-Dimensional (Au-2D) grating. As noted respect to bio-analytes—such a sensor platfor with a Gold, 2-Dimensional (Au-2D) grating. As noted respect to bio-analytes—such a sensor platform has been
previously, the microfluidic flow channel overlies the Au-2D 35 shown to work well in both a flow-through mode as previously, the microfluidic flow channel overlies the Au-2D 35 shown to work well in both a flow-through mode as well as grating in this illustrative sensor structure.
a batch mode (single injection of solute) since its

With continued reference to FIG. 2, it is noted that the sensitivity does not require an accumulation of large layer-pair stack is shown including eight layer-pairs. Advantaneously, structures according to the present disc or greater number of layer-pairs. Additionally, it may be sions through the effect of the 2D grating which is illustra-
observed that an individual Au layer and an individual $A1_2O_3$ tively fabricated from a thin Au lay observed that an individual Au layer and an individual Al_2O_3 tively fabricated from a thin Au layer having an array of layer—which together form an individual Au/ Al_2O_3 layer—spaced-apart holes formed therein. The in pair—are illustratively shown as 16 nm and 30 nm thick, which is several hundreds of nanometers in wavelength—is respectively. Advantageously, the thicknesses of the indi- 45 directed onto and through the layers of the HM vidual layers of Au and $A₂O₃$ may range from few nm (2-3) noted previously—illustratively includes 16 individual layto tens of nanometers (30-50) such that the thickness of the ers (8 layer-pairs) of reflectiv

The HMM structure may comprise a multistack of any such light is "concentrated" into a very small volume much metal-dielectric bilayers. More specifically, it is noted that 50 smaller than the wavelength(s) of light. while the individual layers are shown as comprising Au and \overline{A} As this light strikes the HMM, it excites free electrons $A1_2O_3$ respectively, structures according to the present dis-
resulting in their oscillation closure may be fabricated from alternative materials as well. confined propagating surface wave—a surface plasmon
For example, the Au layers may be fabricated from Ag, Pd, polariton. This propagating surface wave in turn Pt, Ti, and doped semiconductors, while the Al_2O_3 layers 55 bulk wave propagating across the sensor structure. The may be fabricated from any other suitable dielectric or oxide presence of the wave(s) results in strong may be fabricated from any other suitable dielectric or oxide presence of the wave(s) results in strong, sharp dips in the material.

As shown further in FIG. 2, a dielectric spacer layer effect. Advantageously, the combination of the surface plas-
overlies the layer-pair stack. This dielectric spacer layer may mon and bulk plasmon waves excited through

diffraction grating (Au-2D) having an average period of 500 techniques: The reflectance of broadband light can be moni-
nm and a hole size of 160 nm. Since the role of the tored for frequency shifts as function of the mole

 \overline{a} , and the set of \overline{a}

circuitry for executing that software to perform the function. diffractive grating is to couple the incoming radiation with The invention as defined by such claims resides in the fact the photonic nanostructure all range a

means are combined and brought together in the manner
which the claims call for. Applicant thus regards any means
which can provide those functionalities as equivalent as
those shown herein. Finally, and unless otherwise e and subsequently sensed by structures constructed according to the present disclosure.

that FIG. 1, a multi-stack hyperbolic metamaterial—com-
that is the present disclosure may be fabricated using well EXECT the grown over micro-glass substrates with Al_2O_3 and Au Turning now to FIG. 2, there is shown a schematic of an 25 pellets used as source materials.

detected enters an input of the microfluidic channel, flows across/within the GC-HMM, and subsequently exits an grating coupled metamaterial.

Illustratively, the metamaterial shown includes a stack of GC-HMM sensor structure. As a result of this contact, a number of the solute molecules are adsorbed onto the surface of the sensor structure. Advantageously, and with

to tens of nanometers (30-50) such that the thickness of the ers (8 layer-pairs) of reflective and conductive gold and metal-dielectric bilayer is deeply subwavelength $(λ /10)$. transparent aluminum oxide and a dielec directed onto and through the layers of the HMM which—as

material.

As shown further in FIG. 2, a dielectric spacer layer effect. Advantageously, the combination of the surface plastens of nanometers.

Overlying the dielectric layer is a 2D subwavelength gold 65 platform may be employed by using multiple read-out diffraction grating (Au-2D) having an average period of 500 techniques: The reflectance tored for frequency shifts as function of the molecular

binding events and/or for angular shifts of narrow banded spin-coating or spray techniques. The percentage of cover-
(almost monochromatic) light.

In the spin - coating or spray techniques. The percentage of cover-

age a

Notably, and as will be readily appreciated by those controlled to couple the incoming radiation with the HMM skilled in the art—it is oftentimes desirable to detect nanostructure. With respect to HMM structures employing molecular entities with great specificity. Advantageously, 5 randomly-dispersed nanoparticles, we note that we have
sensor devices according to the present disclosure may be randomly dispersed 200 nm sized nanoparticles on sensor devices according to the present disclosure may be
made so specific through the use of one or more specific
"trap" molecules. By way of illustrative example only, such
trap molecules may include immobilized enzymes enzyme-substrate combination being detected. The trap mol-
ecules may be so immobilized onto the top layer of the
GC-HMM structure. Specific example of trap molecules, not
exhaustive of all biomolecular interactions, may i

according to aspects of the present disclosure may be nanoparticles also provides a much stronger coupling effect
constructed using an innovative, lithographically free cou-
than a single nanoparticle, as the reflection di pler positioned between incident radiation and the HMM 20 ing to resonant conditions are considerably larger (4-5 structure. More particularly, and as schematically depicted times). in FIG. 3(A), such configurations may include a specific With respect to an HMM exhibiting a randomly dispersed
hybrid partly leaky waveguide (HPLW) overlying the HMM 100 nm TiO₂ nanoparticles with 60% coverage, we stack. Advantageously, with such a configuration, evanes-
cent modes generated by the HPLW allow the coupling with 25 and BPPs when compared to just one nanoparticle. As cent modes generated by the HPLW allow the coupling with 25 and BPPs when compared to just one nanoparticle. As highly confined modes of HMM structure thereby advanta-
shown in that figure, depicting Hz at 1795 nm, we may highly confined modes of HMM structure thereby advanta-
geously resulting in an efficient, ultrathin light coupler/ that the period of SPP propagation is slightly larger than that geously resulting in an efficient, ultrathin light coupler / that the period of SPP propagation is slightly larger than that decoupler.

of just 1 nanoparticle—approximately 1500 nm, correspond-

sensor, we experimentally replaced the earlier described 30 Finally, with respect to a MINI having a randomly disgrating-coupling mechanism with a waveguide coupler persed 100 nm $TiO₂$ nanoparticles with 60% linear using submicron layer of methyl methacrylate (MMA) on a 600 nm Ag grating, we may observe in FIG. 3(I) that coated with a thin (5-15 nm) layer of metal (Au or Pd) (FIG. reflection spectrum for the MINI with the grating, HM 3(B)) or by employing a random distribution of nanopar-
ticles and nanoparticles alone. The ticles acting as "scatterers" to add momentum to the incom- 35 random arrangement of the nanoparticles both with the

sion diagram for transverse magnetic modes (dotted lines) in of large reflection dips. If we further examine the mode a lithography-free HMM system in terms of photon energy 40 around 1915-1920 nm, the quality from the nan a lithography-free HMM system in terms of photon energy 40 around 1915-1920 nm, the quality from the nanoparticles versus momentum and (right) electric field magnitude for alone is very light. But when we couple to the gra

We note that with respect to the MMA coated with a thin As those skilled in the art will readily appreciate, a planar metal, biomolecular detection studies with these alternative 45 waveguide can generate an evanescent fie hydrogen gas detection. By using Pd as the top metal layer, couplers to an underlying hyperbolic nanostructure. Advan-
which can absorb H₂ from the surrounding medium, altering tageously, our lithography-free structures its dielectric properties, we can measure the shift $\Delta\lambda$ of the 50 HMM mode in response to particular concentrations of H₂. HMM mode in response to particular concentrations of H_2 . cally improve the cost-effectiveness of sensor systems
These shifts are large, reproducible, and completely revers-
ible when H_2 is removed from the environme we also have preliminary theoretical results for the lithog-

Turning now to FIGS. 4(A) and 4(B), there is shown a top

raphy-free system that validate the novel optical coupling 55 view and side view respectively of yet a mechanism between incident radiation and the BPP modes embodiment of an HMM sensor structures according to the
in the HMM. The mode dispersion diagram (FIG. 3(D) left) present disclosure. As may be observed from those figu of the system with an Au top layer (in the absence of a series of channels are nanofabricated across the MINI
functionalization and captured analytes) shows the presence nanostructure such that an analyte fluid reservoir (functionalization and captured analytes) shows the presence nanostructure such that an analyte fluid reservoir (shown in of transverse magnetic modes that can be excited by incident 60 the base of a chip) is connected to a photons from the superstrate, corresponding to sharply-
defined minima in reflectance. The electric field profiles for
diameter(s) can advantage ously range from few tens of defined minima in reflectance. The electric field profiles for diameter (s) can advantageously range from few tens of three of these modes (FIG. $3(D)$ right) illustrate the coupling nanometers to 500 nm such that a biolog three of these modes (FIG. 3(D) right) illustrate the coupling nanometers to 500 nm such that a biological sieve is
of the incoming radiation to BPP excitations. produced thereby filtering large untargeted analytes. More

Large area lithography-free sensing areas were realized 65 by depositing 100 and 200 nm TiO₂ nanoparticles with 60% and 80% coverage. The nanoparticles can be deposited by

biotinylated polymers, etc.—among others. and at each of these wavelengths, we visualize strong bulk
Alternative embodiments of HMM sensor structures mode coupling. Interestingly, the random distribution of Alternative embodiments of HMM sensor structures and coupling. Interestingly, the random distribution of cording effect

coupler.
To explore the potential of a lithography-free HMM ing to an added momentum of 0.0042 nm^{-1} .

reflection spectrum for the MINI with the grating, HMM with grating and nanoparticles, and nanoparticles alone. The ing light and to match the momenta of the bulk plasmon
polaritons (FIG. 3(C)).
FIG. 3(D) shows (left) a theoretically calculated disper-
caused by the nanoparticles and allows for only discrete sets FIG. 3(D) shows (left) a theoretically calculated disper-caused by the nanoparticles and allows for only discrete sets sion diagram for transverse magnetic modes (dotted lines) in of large reflection dips. If we further ex three modes located at A, B, C in left plot—according to we find it is quite enhanced. The magnetic field plot is shown aspects of the present disclosure. $\frac{1}{2}$ in FIG. 3(J).

tageously, our lithography-free structures require—as their name suggests—no lithography, and therefore may dramati-

produced thereby filtering large untargeted analytes. More specifically, this filtration may be enhanced by nanostructuring a reticulate "Cheval de Frise"—or alternative structures—at the bottom of the channel.

FIG. 5 is a photograph of a GC-HMM sensor device disclosure may be advantageously integrated into larger according to the present disclosure integrated with a micro-
fluidic channel and sample tubing. As depicted in that F fluidic channel and sample tubing. As depicted in that FIG. manner, analytes detected by the structures according to the 5, a solution including material(s) to be detected, enters the present disclosure may be collected, o microfluidic channel from one of the tubes and exits the s and/or reported in an alternative usable form(s). Outputs channel through the other tube. As previously noted, as the s and/or reported in an alternative usable fo thereby permitting detection of the solute(s) by the mecha-
nism(s) described above.
appended hereto.

With reference to FIG. 6, there is shown a plot of 10

permittivity vs. wavelength of Au/Al_2O_3 HMM determined

using effective media theory which shows a hyperbolic
 $\frac{1}{a}$. An optical sensor platform comprising:
 $\$ dispersion at 520 nm.
EIG 7 is a plot showing reflectorse vs. weylength for an analyte reservoir overlying the substrate;

FIG. 7 is a plot showing reflectance vs. wavelength for an analytic reservoir overlying the substrate;
a hyperbolic metamaterial (HMM) stack overlying the illustrative GC-HMM at various angles of incidence. As 15 a hyperbolic metamaterial may be observed from this plot, the GC-HMM sensor may be observed from this plot, the GC-HMM sensor
at a dielectric overlying the HMM stack; and
a dielectric overlying the HMM stack; and structure shows four prominent reflectance dips, correspond a different overlying the HMM stack, and
in the hull plasman polaritan modes, and two work reflectors and optical structure overlying the dielectric; ing to bulk plasmon polariton modes, and two weak reflectrical modes over an optical structure overlying the dielectric and optical structure the structure overlying the dielectric structure expansion of the structure wher tance minima in shorter wavelengths, corresponding to the
surface plasmon polariton modes. A blue shift in resonance 20
wavelength with increasing angle of incidence indicates all
six modes are guided modes.

ciate that while the methods, techniques and structures optical energy is directed to the HMM via the optical
according to the present disclosure have been described with as structure a detectable shift in reflected optica according to the present disclosure have been described with 25 structure a regnect to perticular implementations and/or embedimentations 25 produced. respect to particular implementations and/or embodiments, $\frac{1}{2}$. The optical sensor platform according to claim 1 those skilled in the art will recognize that the disclosure is not so limited.

that multiplexing assays (assays that simultaneously mea- 30 wherein the optical structure includes a sub-incron sure multiple analytes in a single assay run) are a very method in method with a layer of metal.
4. The optical sensor platform according to claim 3 important component of contemporary sense and measure-
ment protocols. Of particular significance, sensor structures $\frac{1}{2}$ according to the present disclosure exhibit an inherent $\frac{1}{2}$. The optical sensor platform according to claim 4 according to the present disclosure exhibit an inherent structure in the metal is one selected multiplexing functionality as they are based in large part on 35 wherein the metal is of Pd, and Pt.

disclosure simultaneously evaluate an overall wavelength wherein the optical structure includes a random distribution of $\frac{1}{\text{minimize}}$ of nanoparticles. shift of all modes to discriminate binding events of small $\frac{01}{2}$. The optical sensor platform according to claim 6 molecules—which can be detected only from the most $\frac{40}{40}$ 7. The optical sensor platform according to claim 6
sensitive modes, with recreat to highling quanta of large wherein the random distribution of nanoparticles sensitive modes—with respect to binding events of large wherein the random distribution of nanoparticles includes
molecules that may be detected by all modes. By evoluting $\overline{102}$ nanoparticles exhibiting a diameter of molecules that may be detected by all modes. By exploiting this sensitivity and intrinsic multimodal selective response, this sensor structures according to the present disclosure are able
to provide an extremely sensitive biosensing platrorm for 45 manoparticles provide at least a 60% linear coverage of the biological samples—to detect ultra-low molecular weight
anaytes. Further theoretical discussion(s) of our HMM sens-
ing structures is provided in the Appendix attached hereto. Lastly, it is noted that structures according to the present $* * * * * *$

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- the sensor platform configured such that when incident At this point, those skilled in the art will readily appre-
the sensor platform configured such that when incident
optical energy is directed to the HMM via the optical

wherein the optical structure includes a grating.
3. The optical sensor platform according to claim 1

In particular, those skilled in the art will readily appreciate 3. The optical sensor platform according to claim 1
at multiploxing assays (assays that simultaneously mea, 20) wherein the optical structure includes a sub-m

the different sensitivity of the bulk plasmon polariton modes.
More particularly etrustives according to the present $\overline{6}$. The optical sensor platform according to claim 1 More particularly, structures according to the present **6.** The optical sensor platform according to claim 1 velocity explored wherein the optical structure includes a random distribution

nm .