METHOD AND APPARATUS USING VOLUME HOLOGRAPHIC WAVELENGTH BLOCKERS

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ABSTRACT
The invention disclosed here teaches methods to fabricate and utilize a non-dispersive holographic wavelength blocker. The invention enables the observation of the Raman signal near the excitation wavelength (~9 cm⁻¹) with the compactness of standard thin film/holographic notch filter. The novelty is combining several individual volume holographic blocking notch filter (VHBF) to form one high optical density blocking filter without creating spurious multiple diffractions that degrade the filter performance. Such ultra-narrow-band VHBF can be used in existing compact Raman instruments and thus will help bring high-end research to a greater number of users at a lower cost.

31 Claims, 9 Drawing Sheets
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FIG. 3

FIG. 4
FIG. 5A

FIG. 5B
FIG. 8

FIG. 9
FIG. 11
METHOD AND APPARATUS USING VOLUME HOLOGRAPHIC WAVELENGTH BLOCKERS

CROSS-REFERENCE TO RELATED APPLICATION

The present patent application is a continuation and claims the priority benefit of U.S. patent application Ser. No. 12/315, 470 filed Dec. 3, 2008, which claims the priority benefit of U.S. provisional patent application No. 61/137,871 filed on Aug. 4, 2008, the disclosures of which are incorporated by reference herein in their entirety.

BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention relates to a method and apparatus for fabricating and using volume holographic wavelength blockers of high optical density and narrow bandwidth. Wavelength blockers are used to attenuate the signal of a pump source, such as lasers, while letting a scattering signal such as, but not limited to, fluorescence or Raman to go through. Thick reflective volume holographic elements (>typ. 0.1 mm thickness) have narrow rejection band but have limited attenuation of the order of optical density of 1 to 2. It is desirable to have a narrow spectral band rejection in conjunction with high attenuation reaching at least an optical density of 6 for Raman spectroscopy for example.

2. Background Art

Wavelength blockers, also called notch rejection filters, are an essential component in Raman and fluorescence instruments. The purpose of the wavelength blocker is to greatly attenuate the backscattered light from the laser illuminating a sample under test, while letting the faint Raman spectrally shifted signal pass through. Two non-dispersive filter technologies are currently used for the wavelength blocker: holographic and thin film. Commercial holographic notch filter technology uses holographic recording in a thin film of dichromated gelatin to produce a notch filter with 3 dB bandwidth of 350 cm\(^{-1}\) and optical density of 6. Commercial thin film technology uses deposition of many layers to obtain a 3 dB bandwidth of approximately 600 cm\(^{-1}\) and optical density of 6. Both technologies provide a compact size wavelength blocker element with a 10 mm aperture diameter and several millimeters thickness. However, both notch filter technologies are limited to observing Raman spectral shift above approximately 350 cm\(^{-1}\).

The Raman signal in the low frequency shift region, i.e., near the frequency of the excitation laser, contains critical information about the molecular structure. For example, carbon nanotubes exhibit vibration modes in the range of 150 cm\(^{-1}\) to 200 cm\(^{-1}\) depending on their size. Relaxation in liquids, solutions and biological samples exhibit Raman shift in the range between 0 and 400 cm\(^{-1}\). U.S. Pat. Nos. 5,684, 611 and 5,691,989 describe the use of reflective volume holographic filters (VHG) with millimeters thickness as filters producing 3 dB bandwidth of the order of 10 cm\(^{-1}\). VHGs produced in a glass material are now commercially available and show long lifetime, high efficiency and excellent transmission in the red and near infrared. The photosensitive glass can contain, for example, silicon oxide, aluminum oxide and zinc oxide, fluorine, silver, chlorine, bromine and iodine, cerium oxide. Composition and processes for manufacturing the photosensitive glass are described in U.S. Pat. No. 4,057, 408, the disclosure of which is incorporated herein by reference. Large area (30x30 mm) reflective VHGs are restricted to the millimeter range thickness due to the material absorption. The optical density (O.D) achievable is therefore limited to O.D near unity (i.e., ~90% efficiency) with thickness of 1.5 mm and transmission of 97% to 98% away from the notch in the near infrared.

By carefully individually aligning a cascade of VHGs, researchers have shown that the optical density can be added up: a cascade of 4 VHGs with each exhibiting an optical density of one yields a compound notch with an optical density of 4. Commercial instruments comprising individual alignment fixtures for each VHG exhibit an optical density ranging from 4 to 6 with bandwidth of 10 cm\(^{-1}\). However, there are several drawbacks to this approach:

1. The alignment procedure is complicated and required for each VHG separately.
2. The footprint is large (~100 cm\(^3\)) and as such not suitable to replace standard notch filters in existing Raman instruments.
3. The surface of each VHG contributes to Fresnel reflection loss.
4. Upon rotation of the assembly, the individual VHGs spectrally shift at different rates, thus reducing severely the optical density and broadening the overall blocker bandwidth.

The technology utilized to observe the Raman signal close to the laser excitation (~9 cm\(^{-1}\)) is based on cascading dispersive spectrometers. The cascaded spectrometers are bulky (~1 m\(^3\)), expensive (~$100K) and of moderate transmission (~50%).

SUMMARY OF THE INVENTION

The invention disclosed here teaches methods to fabricate and utilize a non-dispersive holographic wavelength blocker to overcome all the limitations outlined above. The invention enables the observation of the Raman signal near the excitation wavelength (~9 cm\(^{-1}\)) with the compactness of standard thin film/holographic notch filter. The novelty is contacting several individual volume holographic blocking notch filter (VHBF) to form one high optical density blocking filter without creating the spurious multiple diffractions that yield unacceptable rejection ratios. Such ultra-narrow-band VHBF can be used in existing compact Raman instruments and thus will help bring high-end research to a greater number of users at a lower cost.

BRIEF DESCRIPTION OF THE DRAWINGS

These and other features, aspects and advantages of the present invention will become better understood with regard to the following description, appended claims, and accompanying drawings where:

FIG. 1: Grating wave vector representation of four slanted reflective VHBF diffracting the same wavelength.

FIG. 2A, 2B: Illustration of stacked reflective VHBF assembly.

FIG. 3: Illustration for tuning the Bragg wavelength of each VHG in the VHBF assembly.

FIG. 4: Plot of a typical angular selectivity of one VHG in the VHBF stack.

FIG. 5A, 5B: Spectral response of a VHBF assembly showing addition of optical densities.
For the following analysis, we will assume that the collimated incident beam \( \mathbf{311} \) propagates in the direction of the \( z \)-axis: \( \mathbf{k}_{\text{inc}} = \mathbf{k}_{\text{inc}} \). We will allow a grating slant \( \phi \) (angle between grating vector and surface normal) only in the \( x \)-\( z \) plane. We assume that the illumination is of single frequency. The laser wavelength is chosen slightly below the normal incidence wavelength of each VHG in the stack.

Following the illustration in FIG. 3, the first VHG is positioned with its grating vector \( \mathbf{K} (310) \) in the \( x \)-\( z \) plane and rotated around the \( x \)-axis to fulfill the Bragg condition according to equation (1). The facet normal of the first VHG defines the incidence angle \( \Theta_{\|} \) of the entire stack with respect to the collimated illumination direction \( \mathbf{k}_{\text{inc}} \). The orientation of the facet normal of subsequent VHGs with respect to the incident beam, i.e., \( \Theta_{\|} \) are collinear with each other since we assume the VHG in the stack are in mechanical contact.

For the subsequent VHGs after the first one, fine wavelength tuning is achieved by rotating the VHG around its surface normal, the only degree of freedom left, by an angle \( \Theta_{\perp} \).

Using Snell's law, the incident beam wave vector in the material is:

\[
\mathbf{k} = \begin{pmatrix}
\cos(\Theta_{\|})
\sin(\Theta_{\|})
\sin(\Theta_{\perp})
\end{pmatrix}
\]

where \( \Theta_{\|} \) is the angle between \( z \)-axis and \( \mathbf{K} \) and \( \Theta_{\perp} \) the angle between surface normal and \( \mathbf{K} \) measured inside the medium. After rotation of the VHG around the \( x \)-axis by an angle \( \Theta_{\perp} \) and around the surface normal by angle \( \omega \), the VHG's grating vector \( \mathbf{K} \) is:

\[
\mathbf{K} = \mathbf{k} \cos(\omega) \sin(\phi) + \sin(\Theta_{\perp}) \cos(\omega) \cos(\phi)
\]

Using \( \cos(\Theta_{\perp}) = \mathbf{K}/(k\mathbf{K}) \) and equation (3), we find the notch wavelength \( \lambda_{\text{not}} \) as a function of the angles \( \omega \) and \( \Theta_{\perp} \):

\[
\lambda_{\text{not}} = \lambda_{0} \cos(\phi) \sin(\Theta_{\perp}) \sin(\Theta_{0} \sin(\Theta_{0} \sin(\phi)))
\]

From equation (4), we observe that individual VHGs can be Bragg-matched to the required notch wavelength by adjusting the rotation angles \( \omega_i \) for each grating \( i=2, \ldots, N \). The fine wavelength tuning is only possible when \( \Theta_{\perp} \approx 0 \).

A typical angular selectivity curve for an individual VHG is given in FIG. 4. The angular 3 dB bandwidth is 0.4 degrees. In another embodiment, the slant angle of each VHG is chosen such that the diffracted beams do not satisfy the Bragg condition for all other VHGs. From the measurement shown in FIG. 4, a value of at least 1 degree for the slant angle has been selected to satisfy that condition.

The rejection ratio of the VHBF assembly is the compounded rejection of each VHG in the stack when the alignment procedure outlined in the embodiments above is followed. This is justified because there are no coherent effects between the diffracted beams with the arrangement of the grating wave vector of each VHG described above. An example of spectral response of the notch filter with the VHBF assembly of one and three individual VHBF is shown respectively in FIGS. 5A and 5B.
We prepared six individual reflection VHGs with thickness of 1.6 mm and diffraction efficiencies near 90% (corresponding to optical density near unity). Anti-parallel diffraction wavelength and slant angles are given in table 1.

In one embodiment, each of the successive five VHGs is brought into direct mechanical contact to the previous VHG. After alignment, individual gratings are secured to the stack by an index matching epoxy. This procedure ensures that the internal incident angle $\Theta_{s}$ is the same for every grating in the stack. Only the rotation angle $\phi_{r}$ is used to fine tune the Bragg wavelength.

The laser used for the alignment is a wavelength locked semi-conductor laser diode at 785.0 nm, which is subsequently ASE filtered by a slanted reflection VHG. Grating #1 is aligned for Bragg diffraction with $\phi_{r}$=0 and $\Theta_{s}$=2.7 deg.

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Now, let’s determine what happens when the stack of bonded VHGs is wavelength tuned.

In another embodiment, wavelength tuning is performed by varying the incident angle from the initial alignment angle $\Theta_{s}$ to a new incident angle $\Theta_{s} + \Delta \Theta_{s}$. For all VHGs in the stack, the new notch wavelength will vary according to equation (4) and the difference in wavelength between any two gratings can be computed to be:

$$\Delta \lambda = \left( \lambda_{s} \cos(\phi_{r}) - \lambda_{s} \cos(\phi_{r}) \right) \sin(\Delta \Theta_{s})$$

(5)

Note that the wavelength shift between any two gratings does not depend on the rotation terms $\phi_{r}$. This is due to the constraint that at the alignment angle $\Theta_{s}$ of the stack, the wavelength shift $\Delta \lambda$ is equal to zero.

Table 1 gives a standard deviation of 0.069 nm for the quantity $(\lambda_{s} \cos(\phi_{r}) - \lambda_{s} \cos(\phi_{r}))$. The stack of six VHGs was aligned at a value for $\Theta_{s}$ of 2.7 degrees and tuned by $\Delta \Theta_{s}$ of 11.4 degrees (these are values inside the material of index n=1.5). According to equation 5, we expect to observe a broadening of the overall bandwidth by 0.29 nm. The experimental result is shown in FIG. 6A. As expected, the 6-stack wavelength blocker maintains a single transmission notch at all tuning angles. The measured spectral bandwidth broadening is half the computed value (0.14 nm vs. 0.29 nm).

Light transmission of the six-stack wavelength blocker is measured by a CARY 500 spectrometer. The transmission measurement in FIG. 6B shows that the 9.6 mm thick filter stack (6 times 1.6 mm) transmits greater than 80% of the incident light outside the notch. The first and last VHG facets are without anti-reflection (AR) coatings. An additional 8% transmission could be gained by adding an AR coating to the outside facet of the first and last VHG in the stack.

In another embodiment, the Raman excitation laser light source is a laser whose amplified spontaneous emission is filtered as illustrated in FIG. 7A. A laser light source 700 is collimated by collimating assembly 710. A slanted reflective VHG 720 is positioned to receive the collimated beam. The diffracted beam 730 is the ASE filtered beam. The specularly reflected light beam 740 is propagating in a different direction. In other embodiments, more than one ASE filter can be used to further reduce the ASE content of the laser.

FIG. 7B shows the spectrum of the unfiltered and filtered laser diode measured with an ANDO double spectrometer with 0.05 nm resolution. We observe that the ASE of the original laser diode is drastically reduced. The spectrometer distorts and broadens the actual ASE filtered spectrum due to stray light inside the spectrometer. The optical density of the fabricated stack is measured at 780.7 nm with an ASE filtered signal at the frequency laser light source.

The collimated light beam of dimension 1 mm x 2 mm is incident on the wavelength blocker. The transmitted light is fiber coupled to a multimode fibre and sent to the spectrometer. The result is shown in FIG. 8. An attenuation of the laser power of 60 dB, corresponding to an optical density of 6, is achieved. The stack was assembled at a wavelength of 785.1 nm. We have shown that after tuning the stack by 5 nm, an optical density of 6 was maintained.

Another embodiment in the invention is a means to angularly tune the VHFBS assembly so that the Bragg wavelength of the VHBF always tracks the wavelength of the excitation laser in order to obtain maximum optical density (maximum rejection of the excitation light). An example of a tuning mechanism consists of positioning the VHBF on a rotation stage and rotating the stage. A detector is added to receive a portion of the attenuated pump after the VHBF assembly. The signal can be used as feedback to the tuning mechanism. FIG. 9 illustrates the tuning and feedback mechanism. The VHBF 905 is placed on the rotation stage 910. The collimated signal 900 is incident on the VHBF 905. A fraction of the transmitted beam 930 is deflected by the beam-splitter 925 and directed to a photodetector 920. The electrical signal is processed by a computer or microprocessor 915 and a feedback signal is sent to the rotation stage to minimize to photodetected power.

Another embodiment is an apparatus that uses the VHBF assembly of the embodiments above as illustrated by FIG. 10. A laser source 1003 is collimated and ASE filtered by the assembly 1004. The ASE filtered beam 1005 is reflected by a dichroic beam-splitter 1010 towards a lens assembly 1000 that focuses the laser beam onto a sample under examination. The dichroic beam-splitter 1010 reflects the laser beam and is transparent to other wavelength. In yet another embodiment, the dichroic beam-splitter 1010 may be a reflective or transmissive VHGF or any other type of narrowband filter. The signal generated from the sample as a result of the excitation laser beam (fluorescence, Raman) as well as the backscattering of the laser is recollimated by the same lens assembly 1000. The signal is transmitted through the dichroic beam-splitter 1010 and incident on the VHBF assembly 1015 that may also include the tuning assembly disclosed in the embodiment above. Further spatial beam filters may be incorporated in the path of the signal beam to perform a confocal system. After the VHBF assembly, the laser light is rejected and the Raman, fluorescence or any other signal generated by the excitation laser impinges on a dispersive element 1025 such as, but not limited to, a diffraction grating. The speckle dispersed signal is then received by an array of photodetector 1020. The array of photodetectors can be one or two dimensional.
In another embodiment, many of the discrete functions that comprise a standard Raman or fluorescence system, such as laser, ASE filtering, dichroic beam-splitters and wavelength blocker are integrated in a single holographic glass wafer. FIG. 11 illustrates the embodiment. A laser diode 1100 is collimated to produce collimated beam 1115 which is directed to the entrance facet of a holographic glass wafer 1110. A grating 1120, recorded holographically using a transmission geometry, filters the collimated beam 1115 and directs it to an identical grating 1130, also recorded holographically using a transmission geometry. The role of the grating 1130 is of an ASE filter and dichroic beam-splitter. The ASE filtered beam is then brought to a focus by a lens assembly 1150. The wavelength blocker is a cascade of VHG s 1140 whose grating vector amplitude and direction are designed to diffract the same wavelength. The VHG s 1140 are recorded holographically with the transmission geometry. The wavelength blocker attenuates the backscattered laser excitation light. The dimension of the holographic wafer is approximately 10 mm by 15 mm and comprises three distinct functions: ASE filtering, dichroic beam-splitter and wavelength blocker.

After the wavelength blocker, a lens assembly 1150 is used in conjunction with an aperture 1160 to perform confocal measurements. The lens assembly 1150 can be, but is not limited to, a cylindrical lens. A compact spectrometer is built in one glass block, which has a cylindrical surface 1161 to collimated the signal to direct it to a dispersive grating 1162. The spectrally dispersed signal is then capture by an array of photodetectors 1163.

In another embodiment illustrated in FIG. 12, the laser 1200 is used to pump a doped glass region 1210 (for example, but not limited to, Neodymium) which is surrounded by two holographically written reflective VHG s 1205 and 1215 that serve as resonators to amplify the doped glass region and provide laser light.

What is claimed is:

1. A volume holographic wavelength blocking filter assembly comprising:
   a plurality of reflective volume holographic gratings having a cumulative optical density of at least two, each reflective volume holographic grating having a distinct slant angle; and
   a distinct grating spacing, wherein the plurality of reflective volume holographic gratings produce a filtered optical signal when disposed in a path of an optical signal.

2. The volume holographic wavelength blocking filter assembly of claim 1, wherein the optical signal is a Raman signal.

3. The volume holographic wavelength blocking filter assembly of claim 1, wherein the plurality of reflective volume holographic gratings have a cumulative optical density of at least four at the Bragg wavelength.

4. The volume holographic wavelength blocking filter assembly of claim 1, wherein at least two of the reflective volume holographic gratings are bonded with an index matching epoxy.

5. The volume holographic wavelength blocking filter assembly of claim 1, wherein at least two of the reflective volume holographic gratings are secured to each other at their edges and have an index matching fluid between the gratings.

6. The volume holographic wavelength blocking filter assembly of claim 1, wherein at least two of the reflective volume holographic gratings are secured to each other via optical contacting.

7. The volume holographic wavelength blocking filter assembly of claim 1, wherein at least two of the reflective volume holographic gratings have index matched optical coatings and are secured to each other at their edges.

8. The volume holographic wavelength blocking filter assembly of claim 1, wherein at least two of the reflective volume holographic gratings are coated with an anti-reflection material.

9. The volume holographic wavelength blocking filter assembly of claim 1, wherein at least two of the reflective volume holographic gratings are coated with an anti-reflection material.

10. The volume holographic wavelength blocking filter assembly of claim 1, wherein the product of the cosine of the slit angle and the grating spacing for each reflective volume holographic grating forming the stack is substantially equal to each of the other products.

11. The volume holographic wavelength blocking filter assembly of claim 1, wherein each reflective volume holographic grating is successively aligned by a first rotation of a common angle with respect to a collimated single frequency laser beam and a second rotation around the surface normal of each reflective volume holographic grating by an angle to maximize the optical density at the common Bragg wavelength.

12. The volume holographic wavelength blocking filter assembly of claim 1, wherein the slant angle and the grating spacing is measured for each volume holographic grating, each volume holographic grating being passively aligned.

13. The volume holographic wavelength blocking filter assembly of claim 1, wherein the blocking wavelength is tuned by rotation.

14. The volume holographic wavelength blocking filter assembly of claim 1, wherein the blocking wavelength is at least partially tuned by temperature.

15. The volume holographic wavelength blocking filter assembly of claim 1, wherein the holographic material is made of photosensitive glass.

16. The volume holographic wavelength blocking filter assembly of claim 15, wherein the photosensitive glass comprises at least one compound selected from the group consisting of silicon oxide, aluminum oxide, and zinc oxide.

17. The volume holographic wavelength blocking filter assembly of claim 16, wherein the photosensitive glass further comprises an alkali oxide, fluorine, silver and at least one compound selected from the group consisting of chlorine, bromine, and iodine.

18. The volume holographic wavelength blocking filter assembly of claim 16, wherein the photosensitive glass further comprises cerium oxide.

19. The volume holographic wavelength blocking filter assembly of claim 16, wherein the entire filter assembly forms a compact device that is used in a handheld device that is easily transportable.

20. The volume holographic wavelength blocking filter assembly of claim 16, wherein the entire filter assembly is formed on a single photosensitive glass wafer to form a compact device that is used in a handheld device that is easily transportable.

21. An apparatus for Raman spectroscopy, the apparatus comprising:
   a laser, an output of the laser being directed toward physical matter to cause Raman scattering, thereby producing a Raman signal;
   a plurality of reflective volume holographic gratings having a cumulative optical density of at least two, each reflective volume holographic grating having
a distinct slant angle; and
wherein the plurality of reflective volume holographic
gratings produces a filtered Raman signal when dis-
posed in a path of a Raman signal;
the apparatus further comprising an optical spectrometer
disposed in a path of the Raman signal to measure a
spectrum of the Raman signal and to generate a detec-
tion signal; and
a microprocessor to receive the detection signal to deter-
mine properties of the physical matter.
22. The apparatus of claim 21, further comprising:
a beam-splitter positioned in the path of the filtered Raman
signal to sample a portion of the beam; and
a photodetector, wherein the sampled portion of the beam
is detected by the photodetector to generate a signal to
create a feedback mechanism to tune the laser wave-
length to maximize the optical density at the Rayleigh
wavelength.
23. The apparatus of claim 21, further comprising a filter
assembly positioned in the path of the laser that removes at
least amplified spontaneous emission from the laser output.
24. The apparatus of claim 23, wherein the filter assembly
comprises one or more volume holographic gratings.
25. The apparatus of claim 21, wherein the blocking wave-
length is tuned by rotation.
26. The apparatus of claim 21, wherein the blocking wave-
length is at least partially tuned by temperature.
27. The apparatus of claim 21, further comprising a fiber
optic cable to deliver the signal to the spectrometer.
28. The apparatus of claim 21, wherein the entire filter
assembly is formed on a single photosensitive glass wafer to
form a compact device that is handheld and easily transport-
able.
29. The apparatus of claim 28, wherein the laser is also
mounted on the single photosensitive glass wafer.
30. The apparatus of claim 21, wherein a volume holo-
graphic grating is used as a dichroic beam-splitter to reflect-
ing the main laser line and transmit other spectral com-
ponents.
31. The apparatus of claim 21, wherein a volume holo-
graphic grating is used to:
direct more than 50% of the laser light toward the sample;
divert ASE light or parasitic longitudinal modes away from
the laser;
direct more than 50% of the Rayleigh scattered light away
from the detection system; and
transmit the Raman scattered light toward the detection
system.