Video Article

Terahertz Microfluidic Sensing Using a Parallel-plate Waveguide Sensor

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Abstract

Refractive index (RI) sensing is a powerful noninvasive and label-free sensing technique for the identification, detection and monitoring of microfluidic samples with a wide range of possible sensor designs such as interferometers and resonators [1,2]. Most of the existing RI sensing applications focus on biological materials in aqueous solutions in visible and IR frequencies, such as DNA hybridization and genome sequencing. At terahertz frequencies, applications include quality control, monitoring of industrial processes and sensing and detection applications involving nonpolar materials.

Several potential designs for refractive index sensors in the terahertz regime exist, including photonic crystal waveguides [3], asymmetric split-ring resonators [4], and photonic band gap structures integrated into parallel-plate waveguides [5]. Many of these designs are based on optical resonators such as rings or cavities. The resonant frequencies of these structures are dependent on the refractive index of the material in or around the resonator. By monitoring the shifts in resonant frequency the refractive index of a sample can be accurately measured and this in turn can be used to identify a material, monitor contamination or dilution, etc.

The sensor design we use here is based on a simple parallel-plate waveguide [6,7]. A rectangular groove machined into one face acts as a resonant cavity (**Figures 1 and 2**). When terahertz radiation is coupled into the waveguide and propagates in the lowest-order transverse-electric (TE_1) mode, the result is a single strong resonant feature with a tunable resonant frequency that is dependent on the geometry of the groove [6,8]. This groove can be filled with nonpolar liquid microfluidic samples which cause a shift in the observed resonant frequency that depends on the amount of liquid in the groove and its refractive index [9].

Our technique has an advantage over other terahertz techniques in its simplicity, both in fabrication and implementation, since the procedure can be accomplished with standard laboratory equipment without the need for a clean room or any special fabrication or experimental techniques. It can also be easily expanded to multichannel operation by the incorporation of multiple grooves [10]. In this video we will describe our complete experimental procedure, from the design of the sensor to the data analysis and determination of the sample refractive index.

Video Link

The video component of this article can be found at http://www.jove.com/video/4304/

Protocol

1. Sensor Design and Fabrication

1. Design a parallel-plate waveguide with one or more integrated cavities (or "grooves"). See **Figures 1** and **2**. Geometry may be based on that given in our previous publications [8,9] or specially designed for the particular application. The following general guiding principles are suggested:
    1. Plate Spacing: In this experiment a plate spacing of 1mm is used for effective coupling to the TE1 mode without the need for special optics. It also ensures single-mode propagation at the frequencies of interest. When using other plate spacings, multimode propagation, dispersion and coupling efficiency should be considered.
    2. Spacers: This plate spacing is maintained using dielectric spacers. Small pieces of glass with very uniform thickness make excellent spacers - in our case, we use shards from a shattered microscope slide, with have a thickness of 1 mm +/- 3 µm.
    3. Plate size: The plates themselves should be wide enough that they can be considered infinite compared to the input beam. (In our case, 4.75 cm for a 1.2 cm beam.) The thickness of each plate must be much thicker than the skin depth, and thicker plates (> 1 cm) are recommended to reduce the possibility of energy passing above or below the waveguide and reaching the detector. Propagation length should be enough that the groove is at least twice its own width away from the input and output faces, but minimized to reduce dispersion.

4. Bottom Plate Geometry: To allow for easy access to the groove, the bottom waveguide plate should be significantly wider than the top plate, while the groove extends almost (but not quite) the entire width of the plate. (See **Figure 1**) This makes it much easier to access the groove and monitor the level of filling.
5. Screws: Both top and bottom plate have an extension so that screws may be inserted to hold the waveguide together without obstructing either the grooves or the propagation path. (See **Figure 1**) The holes in the bottom plate are threaded while top are not.
6. Cavity Geometry: Design for the groove will depend on the desired resonant frequency, the desired linewidth, and the chosen plate spacing, among other factors. It is important to consider the limitations of your fabrication techniques for very narrow or very shallow grooves. Multiple grooves for multichannel sensing have additional requirements [10].
7. Ungrooved Version: a design identical in every aspect WITHOUT a groove should also be fabricated, to be used as a reference.

2. Fabrication of the waveguide can be done by machining. IMPORTANT: do not blunt the edges of the plates, particularly on the input face. Rounded edges are standard practice in many machine shops for safety reasons but a rounded edge on the input face will distort the signal.
3. Assembly Procedure. After the two plates have been fabricated, they should be assembled into the waveguide.
    1. Use an L-bracket or other flat objects to create a structure with two flat surfaces perpendicular to one another. Place the bottom plate on the horizontal surface and press it flush against the vertical surface. Place the dielectric spacers as close to the screw holes as possible (two per screw, one on each side), being careful not to obstruct the groove or to extend beyond the input face.
    2. Carefully place the top plate flush against the vertical surface and slide it down to sit on the bottom plate and spacers. Holding both plates flush against the vertical surface, insert the screws. Screw them down incrementally in an alternating pattern. This procedure leads to a waveguide with a perfectly flat input face and uniform plate spacing.

2. Experimental Apparatus

This protocol assumes the user has access to a transmission-geometry terahertz time-domain spectrometer (in our case, the Picometrix T-Ray 4,000) and is familiar with obtaining time-domain waveforms and Fourier transforming to the frequency-domain.

1. Confocal Configuration. If not already present, four lenses should be introduced into the beam path in a confocal orientation in order to provide a tight focus at the midpoint of the path.
2. Place an aperture at the focal point. The aperture should be large enough to block all radiation from propagating except through the waveguide. The size of the aperture will determine the beam size propagating in the waveguide (in our case, 12 mm).
3. Place waveguide immediately behind the aperture, with the input face in contact with the aperture and with the waveguide propagation axis aligned as closely as possible with the optical axis. The alignment here is critical - reflections, dispersion, variation in the cut-off and resonant frequencies, and other issues may arise due to improper alignment of the waveguide. Use a secure holder to ensure REPEATABLE PLACEMENT.
4. Syringe holder: it is useful to have a structure that holds the syringe in place so that the tip is aligned with the groove. By doing this you can reduce the possibility of mistakes in the filling due to the motion of the syringe in your hands.

3. Sample Preparation

1. Cleaning Procedure: Disassemble the waveguide. Wash both plates of the waveguide thoroughly in an appropriate solvent to remove any residue from the experiment. Blow dry with compressed air. Reassemble as in 1.3.
2. Syringe Preparation. For best results, we recommend using a different syringe for each material to prevent cross-contamination. If this is not possible, the syringe should also be cleaned with the same solvent.
3. Fill syringe to appropriate fill volume with the liquid to be tested. Try to eliminate any bubbles.

4. Experimental Procedure

1. Place the ungrooved reference waveguide in the apparatus as described in (2.3). Take a reference waveform of the ungrooved waveguide, then remove. This is only necessary once every few hr during each experimental session, depending on the long-term stability of the time-domain spectrometer signal.
2. Place clean grooved waveguide in apparatus, as described in (2.3)
3. Take a waveform for the empty grooved waveguide. NOTE: This must be done every time the waveguide is removed and cleaned. The process of removal and disassembly can lead to very small variations in the geometry of the waveguide. These variations will affect the absolute resonant frequency of the empty and filled grooves but not the observed shift; therefore each "full" measurement requires its own "empty" reference to calculate the shift.
4. WITHOUT MOVING THE WAVEGUIDE, put the filled syringe in place in the holder. Slowly fill the groove, keeping watch that the fill is good, with no bubbles or overflow. (How to determine the correct fill amount is described in the Discussion section.) Take another waveform.
5. If the system has more than one groove, continue filling grooves and taking waveforms as desired.
6. Remove waveguide and clean (as in Step 3).
7. Repeat as many times as necessary. For best results, several data sets for each sample are recommended to reduce the error.

5. Representative Results

Data analysis of these waveforms is straightforward and can follow the experimenter's usual techniques for transforming to the frequency domain. Frequency spectra such as those given in **Figure 3** should result. These can be squared and divided by the reference waveform to obtain power transmission spectra such as **Figure 4**. The linewidth and central frequency of the resonances for the empty and full waveguides can be measured from these spectra, or Lorentzian fits can be performed to increase the accuracy.

The resonant shift caused by the liquid is merely the difference between the observed central frequencies of the resonances for the empty and full waveguides. To convert this to a refractive index measurement, the relationship between the shift and the RI must be established. This can done experimentally by following this procedure with samples of known index, or computationally by conducting simulations of the groove filled with samples of known index [9], or analytically using mode-matching techniques [8]. Once a shift vs. RI curve is established, RI measurements of unknown samples can be accurately performed.

There are a few particular errors that may occur during this procedure. Bubbles or mistakes in the filling of the groove can result in noisy or incorrect data, which is why we recommend multiple data sets for each sample material. Another frequent source of error is in the placement of the waveguides. If the reference and sensor waveguides are placed in exactly the same alignment, any reflections or other artifacts will be the same for both and will divide out of the transmission spectrum. If the alignment is slightly off, the reflections will not divide out and ringing will be observed in the transmission spectra (some minor ringing can be seen in **Figure 4**). If it is not desirable to retake the data, it is possible to eliminate this ringing by trimming the time-domain waveform before the reflection appears, but this greatly reduces the spectral resolution and therefore the refractive index resolution is limited as well.

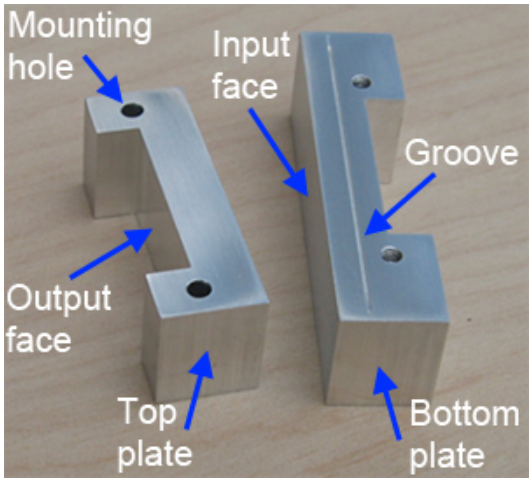

Figure 1. Photograph of the waveguide with relevant parts marked. Note that the groove does not extend the entire length or width of the waveguide and the structure is designed so that the mounting hardware will not obstruct the groove or the path of radiation propagation.

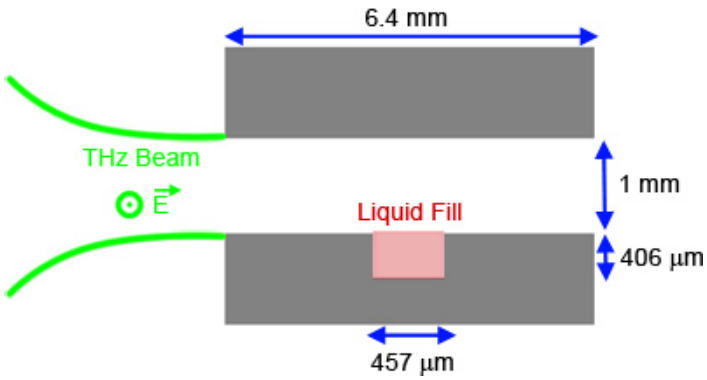

Figure 2. Schematic of the grooved waveguide.

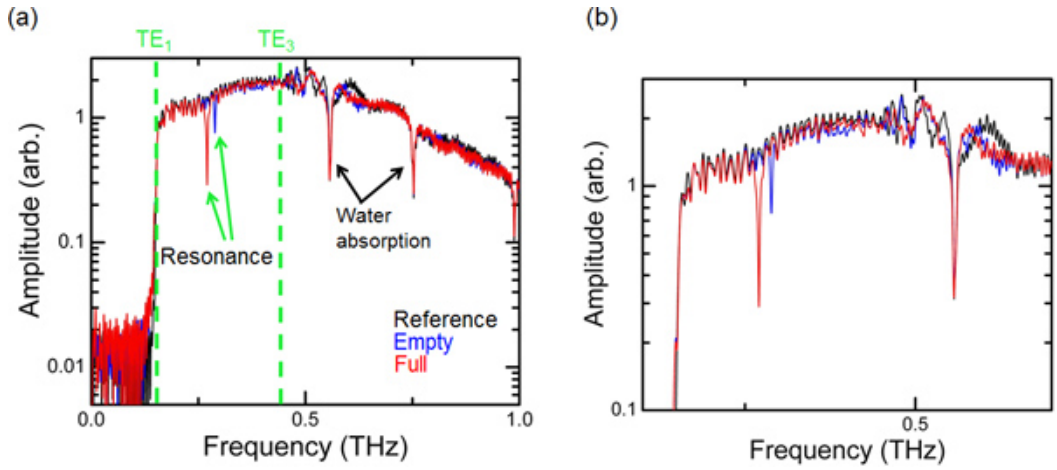

Figure 3. (a) Sample frequency spectra for the reference waveguide (black), the grooved waveguide with no liquid fill (blue), and the grooved waveguide with liquid, in this case tetradecane (red). The cutoff frequencies for the TE_1 and TE3 propagation modes are shown, as are the water vapor absorption lines. **(b)** Closeup of the resonances for the empty and full grooved waveguides.

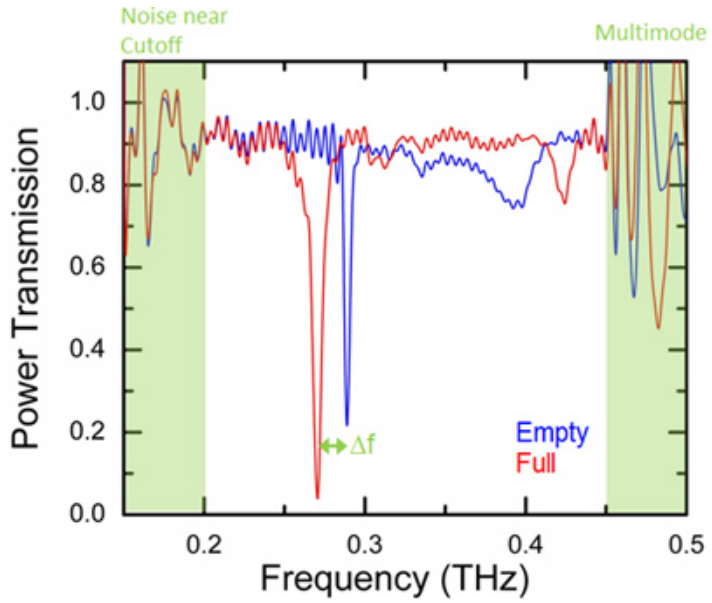

Figure 4. Power transmission spectra for the empty and full grooved waveguides. The difference in frequency between the two resonant features is the resonant shift (Δf), which relates to the refractive index.

Discussion

It should be noted that the refractive index of the liquid under test is determined only at the frequency of the cavity resonance, not over a broad bandwidth. This has a few distinct advantages. First, although our measurements have made use of a broadband terahertz source for characterization purposes, one could also build an equivalent sensing system with a single-frequency THz source with only a limited degree of frequency tunability, an approach that could be much less expensive and more compact. Second, the sensing approach can be parallelized by incorporating multiple grooves into a single waveguide.[10] Each groove would have a slightly different geometry, and therefore a different frequency for sensing. Using a broadband terahertz pulse, one may determine refractive indices (and shifts) independently and simultaneously for multiple liquid samples. This parallel sensing capability would not be easily incorporated into a conventional time-domain terahertz measurement system, in which only a single liquid is measured at a time.

The most important concern with this experimental technique is consistency and repeatability. The assembly and placement of the waveguide and the filling volume can introduce a large amount of error if not consistent. Maintaining a consistent fill volume can be accomplished in a few ways. One, as shown in this procedure, is to use high-precision syringes to measure exact volumes. Another method is to use a laser interferometric system to monitor the actual filling level in the groove [9]. To determine the best syringe volume or fill height, the best results are obtained by gradually filling the groove and monitoring the corresponding shift of the resonant feature. When the groove is full and the liquid

begins to overflow, the resonant feature will be at its lowest frequency. The volume or fill height just before this overflow/saturation point is the best choice and the frequency shift vs. RI response of the device should be calibrated using this value.

There are several other key considerations besides the waveguide assembly and filling volume. Cross-contamination should be avoided through careful cleaning procedures. Evaporation must be considered for lighter molecules and can limit the resolution in these cases. The RI resolution of this procedure in general is limited by the variation between multiple data sets of the same material, but future improvements in the repeatability may reduce the resolution to the limit set by the spectral resolution of the apparatus.

Future improvements for this technique include adapting the sensor design to a closed channel to eliminate filling errors and to allow continuous flow monitoring and developing a reliable cleaning technique that does not require disassembly of the waveguide. There are some limitations that are inherent to the technique - such as the restriction to nonpolar liquids, due to strong terahertz absorption by polar molecules - but others such as the resolution and repeatability have the potential for considerable improvement. As it stands, this technique has been established as a simple and cost-effective technique for RI sensing and monitoring, particularly for industrial applications.

Disclosures

No conflicts of interest declared.

Acknowledgements

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