Video Article

Fabricating Metamaterials Using the Fiber Drawing Method

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URL: https://www.jove.com/video/4299
DOI: doi:10.3791/4299

Keywords: Physics, Issue 68, Optics, Photonics, Materials Science, Fiber drawing, metamaterials, polymer optical fiber, microstructured fibers

Date Published: 10/18/2012

Citation: Tuniz, A., Lwin, R., Argyros, A., Fleming, S.C., Kuhlmey, B.T. Fabricating Metamaterials Using the Fiber Drawing Method. *J. Vis. Exp.* (68), e4299, doi:10.3791/4299 (2012).

Abstract

Metamaterials are man-made composite materials, fabricated by assembling components much smaller than the wavelength at which they operate [1]. They owe their electromagnetic properties to the structure of their constituents, instead of the atoms that compose them. For example, sub-wavelength metal wires can be arranged to possess an effective electric permittivity that is either positive or negative at a given frequency, in contrast to the metals themselves [2]. This unprecedented control over the behaviour of light can potentially lead to a number of novel devices, such as invisibility cloaks [3], negative refractive index materials [4], and lenses that resolve objects below the diffraction limit [5]. However, metamaterials operating at optical, mid-infrared and terahertz frequencies are conventionally made using nano- and micro-fabrication techniques that are expensive and produce samples that are at most a few centimetres in size [6-7]. Here we present a fabrication method to produce hundreds of meters of metal wire metamaterials in fiber form, which exhibit a terahertz plasmonic response [8]. We combine the stack-and-draw technique used to produce microstructured polymer optical fiber [9] with the Taylor-wire process [10], using indium wires inside polymethylmethacrylate (PMMA) tubes. PMMA is chosen because it is an easy to handle, drawable dielectric with suitable optical properties in the terahertz region; indium because it has a melting temperature of 156.6 °C which is appropriate for codrawing with PMMA. We include an indium wire of 1 mm diameter and 99.99% purity in a PMMA tube with 1 mm inner diameter (ID) and 12 mm outside diameter (OD) which is sealed at one end. The tube is evacuated and drawn down to an outer diameter of 1.2 mm. The resulting fiber is then cut into smaller pieces, and stacked into a larger PMMA tube. This stack is sealed at one end and fed into a furnace while being rapidly drawn, reducing the diameter of the structure by a factor of 10, and increasing the length by a factor of 100. Such fibers possess features on the micro- and nano- scale, are inherently flexible, mass-producible, and can be woven to exhibit electromagnetic properties that are not found in nature. They represent a promising platform for a number of novel devices from terahertz to optical frequencies, such as invisible fibers, woven negative refractive index cloths, and super-resolving lenses.

Video Link

The video component of this article can be found at https://www.jove.com/video/4299/

Protocol

Overview

The composite indium/PMMA fiber (**Figure 3**) is produced by drawing a stack of PMMA fibers including a single indium wire (**Figure 2**), which themselves have to be prepared from available PMMA tubes and wires. The steps presented are:

A. Produce a PMMA fiber that contains a single indium wire of diameter appropriate for manual stacking. For this, first prepare a PMMA tube that can accommodate a 1 mm indium wire (Section 1), then include the indium and draw to the required size (Section 2).
B. Stack and draw the obtained individual indium-filled PMMA fibers (Section 3) to the required size.

Sections 4 and 5 detail the drawing processes used in sections 2 and 3.

1. Fabricating the PMMA Jacketing Tube

The PMMA jacketing tube used to structure the 1 mm indium wire is made by stretching and sleeving standard PMMA tubes in the primary draw process (Section 4) to make a final PMMA jacketing tube of ID 1 mm and OD 12 mm.

1. Cut PMMA tubes with ID of 6mm and OD of 12 mm to 600 mm lengths. Several PMMA tubes should be prepared for future use during the sleeving process.
2. Anneal the PMMA tubes in an annealing oven at 90 °C for a minimum of 5 days.
3. Remove one PMMA tube from annealing oven and allow it to cool to room temperature.
4. Clean the surface of the PMMA tube with isopropanol wipes and allow to dry.
5. Attach the PMMA tube to top extender (**Figure 6**) using reflective tape (**Figure 7**).
6. Attach the PMMA tube to primary draw bottom extender (**Figure 6**) using reflective tape (**Figure 8**).

7. Stretch the PMMA tube in the primary drawing process (refer to Section 4). Note that no vacuum is required for this stage. The PMMA tube is stretched from OD 12 mm to 6 mm.
8. Remove the stretched tube from the draw tower after drawing.
9. Cut the stretched tube into 550 mm lengths.
10. Repeat steps 1.3 and 1.4.
11. Heat the top side of the stretched tube with a hot air gun until the material softens and crimp seal the hole using pliers (**Figure 9**).
12. Insert the stretched tube into the new PMMA tube to create the PMMA tube assembly (**Figure 10**). On bottom side of the PMMA tube assembly (*i.e.* the side which has the inner stretched tube open), wrap polytetrafluoroethylene (PTFE) tape as shown in **Figure 10**, to seal the gap between the stretched tube and the new PMMA tube.
13. Attach top end of the PMMA tube assembly (*i.e.* the side which has the inner stretched tube sealed) to the top extender (**Figure 7**), using an inner layer of sticky tape, a middle layer of PTFE tape, and an outer layer of reflective tape. Ensure the PTFE tape is tight and all gaps between the PMMA tube assembly and the top extender are sealed.
14. Attach the PMMA tube to the primary draw bottom extender as shown in 1.6.
15. Stretch and sleeve the PMMA tube assembly in the primary drawing process with vacuum (refer to Section 4). The PMMA tube assembly is stretched from OD 12 mm to 6 mm.
16. The resulting stretched PMMA jacketing tube will have ID/OD of approximately 0.25. Repeat 1.9 to 1.15 until the final PMMA jacketing tube has ID/OD of approximately 0.1 with an ID of 1 mm (**Figure 1**).

2. Fabricating the Indium Filled Fiber

The 1 mm indium wire is sleeved and stretched in the PMMA jacketing tube made in Section 1 using the secondary draw process (Section 5) to produce indium filled fiber with a final OD 1.2 mm.

1. Prepare and anneal PMMA jacketing tubes as shown in 1.1 - 1.4.
2. Cut the indium wire to 550 mm lengths.
3. Insert indium wire into the PMMA jacketing tube to create the indium filled preform assembly as shown in **Figure 11**.
4. Seal the bottom side of the PMMA jacketing tube as shown in 1.11.
5. Attach indium filled preform assembly to the top extender as shown in 1.13 and the secondary draw bottom extender as shown in 1.14.
6. Stretch and sleeve the indium filled preform assembly in the secondary drawing process with vacuum to make indium filled fiber (refer to Section 5) of a final OD 1 mm drawn under 15-20 g tension.
7. Remove the spool of indium filled fiber from the tower after the draw process is finished.
8. Inspect the endface and along the longitudinal length of the indium filled fiber using a light microscope. Problematic defects can include separation between the indium wire and PMMA tubing interface, fluctuations in the wire diameter or fracture cracks along the length of the fiber. Optical microscope images of the indium filled fiber are presented in **Figure 2**, showing a continuous 100 µm indium wire in a 1 mm OD PMMA fiber.
9. Repeat 2.1 to 2.8 until enough indium filled fiber is produced for the indium stacked preform.

3. Fabricating the Indium Stacked Fiber

The indium stacked fiber is fabricated by first stacking the indium filled fibers produced in Secton 2 in a larger PMMA preform jacketing tube, which is then stretched and sleeved to the desired fiber dimensions using the secondary draw process (Section 5).

1. Prepare the PMMA preform jacketing tube as shown in 1.1. For demonstration purposes, we will use a PMMA tube of 12 mm OD and 9 mm ID.
2. Cut the indium filled fibers to 550 mm length.
3. Clean the surface of the PMMA preform jacketing tube and the indium filled fiber with isopropanol wipes and allow to dry.
4. Bundle the indium filled fiber using rubber bands and insert into the PMMA preform jacketing tube, ensuring the fibers are straight and are of a tight fit (**Figure 12**).
5. Anneal the stacked preform assembly in the annealing oven at 90 °C for a minimum of 5 days.
6. Remove the stacked preform assembly from the annealing oven and allow it to cool to room temperature.
7. Attach indium filled preform assembly to the top extender as shown in 1.13 and the secondary draw bottom extender as shown in 1.14.
8. Stretch and sleeve the stacked preform assembly in the secondary drawing process with vacuum to make indium stacked fiber (refer to Section 5). This is stretched to a final OD 0.6 mm drawn under 80 g tension, producing a metamaterial fiber containing 5 mm wires separated by 50 µm. An optical microscope cross-sectional image of the resulting fiber is shown in **Figure 3**.
9. Remove the spool of indium stacked fiber from the tower after the draw process is finished.
10. Inspect the endface and along the longitudinal length of the indium stacked fiber as shown in 2.8 (**Figure 3**).

4. Primary Draw Process

The primary draw process is used to stretch preforms to outer diameters greater than 1 mm. The following procedure is used in Section 1: Fabricating the PMMA Jacketing Tube.

1. Load the preform onto the draw tower by clamping the top extender to the three jaw chuck. Feed the preform into the hot zone of the furnace (**Figure 13**). Align the preform using the XY micrometer stage. Close the top plate of the furnace.
2. The pre-heat stage elevates the temperature of the cross sectional area of the preform to the drawing temperature, using the temperature profile shown in **Figure 14**.
3. Commence the drawing process by increasing the temperature to 185 °C, starting the feed rate at 5 mm/min, draw rate at 6 mm/min and closing the draw unit clamps. Examine the behaviour of draw tension over time (**Figure 15**).

- If the tension increases exponentially, stop the feed and draw units, wait 1 min to allow the preform to heat up to drawing temperature, before starting the feed and draw units again. Repeat the test until the tension stabilizes.
- If the tension falls, increase the draw rate by 1-2 mm/min. Continue increasing the draw rate in 1-2 mm/min increments (as long as the tension either remains constant or starts falling), until the required draw rate is achieved.

4. If vacuum is required, attach the vacuum tube to the vacuum sealed top preform extender using Blu-Tac (**Figure 13**). Turn on the vacuum after the feed and draw units have started to ensure the preform is drawing symmetrically.
5. Use the primary drawing condition in **Table 1** as a guide when drawing the preform. Note the furnace temperature and the ratio between the feed and draw rate have to be monitored to maintain constant OD and the drawing tension. Note that an indicative outer diameter for the drawn fiber can be obtained from a mass balance equation,
$D_{final} = D_{start} (F/D)^{1/2}$
where D_{final} is the final fiber diameter, D_{start} is the initial preform diameter, F is the feed rate, and D is the draw rate. Stop the feeding and drawing rate and switch of the furnace when the preform is finished. Remove the preform from the draw tower once the preform cools to room temperature.

5. Secondary Draw Process

The secondary draw process is used to stretch preforms to ODs smaller than 1 mm. The following procedure is used in Section 2: Fabricating the indium filled fiber and 3: Fabricating the indium stacked fiber.

1. Loading the preform for the secondary draw is the same as in the primary draw process (Step 4.1).
2. The pre-heating stage for the secondary draw is the same as in the primary draw process (Step 4.2).
3. The preform begins to neck-down once the drawing temperature is reached. The drop-down of the preform exits the bottom of the furnace due to the weight of the bottom extender providing the initial drawing force (**Figure 16**).
4. Start the feed rate (2.5 - 5 mm/min) and start increasing the furnace temperature (2.5 - 5 °C) to control the speed of the drop-down. The fiber diameter should be maintained around 250 - 500 µm to prevent the fiber snapping.
5. Attach the fiber to the capstan wheel that is spinning at a slow rate of under 1 m/min initially. Wind the fiber around the dancer wheels and attach to the fiber spool.
6. If vacuum is required attach the vacuum tube as shown in 4.4.
7. The fiber draw will initially be under transient draw conditions. Set the feed rate, draw rate and furnace temperature to the desired draw condition values. Fiber diameter and draw tension will fluctuate until steady state is achieved after a few minutes.
8. Use the secondary drawing condition in Table 2 as a guide when drawing the preform. Note the furnace temperature and the ratio between the feed and draw rate have to be monitored to maintain constant OD and the drawing tension.
9. Stop the process as shown in 4.5.

Representative Results

Metamaterial fibers were produced using the technique described. They was assembled from a preform of 1 mm PMMA fibers containing 100 µm diameter continuous indium wires, shown in **Figure 2**, which in turn had themselves been drawn from a preform of 1 mm indium wires contained inside a 10 mm polymer jacket, which was produced by sleeving appropriately sized polymer tubes, as shown in the schematic of **Figure 1**. A microscope image of the cross section of an example of a metamaterial fiber with plasmonic response in the THz range is shown in **Figure 3**.

The plasmonic response manifests itself such that at low frequencies the material behaves like a metal (low transmission) and at high frequencies like a dielectric (high transmission), with the plasma frequency defining the boundary between the two behaviours. In this specific case, the plasma frequency is expected at 1.2 THz, however our technique permits this to be readily changed by varying the draw speed, which in turn changes the radius and separation of the wires, as presented in Ref.[8]. The resulting high-pass filtering behavior of the metamaterial fiber, for incident THz waves with their electric fields directed along the wires, can be measured via terahertz time domain spectroscopy [11].

Figure 4.i.a shows experimental measurements of this fibre type drawn to three different dimensions. This agrees very well with theory, shown in **Figure 4.i.b.** In both cases the plasma frequency dependence on diameter is apparent. Analysis of the particular fiber shown in **Figure 3** gives the plasmonic response shown in **Figure 4.ii** where the plasma frequency is at 0.6 THz.

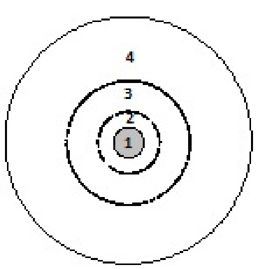

Figure 1. Multiple sleeved jacket cross section schematic with single indium wire. 1 is the indium wire, 2 is the 1st jacketing PMMA tube, 3 is the 2nd, and 4 is the 3rd.

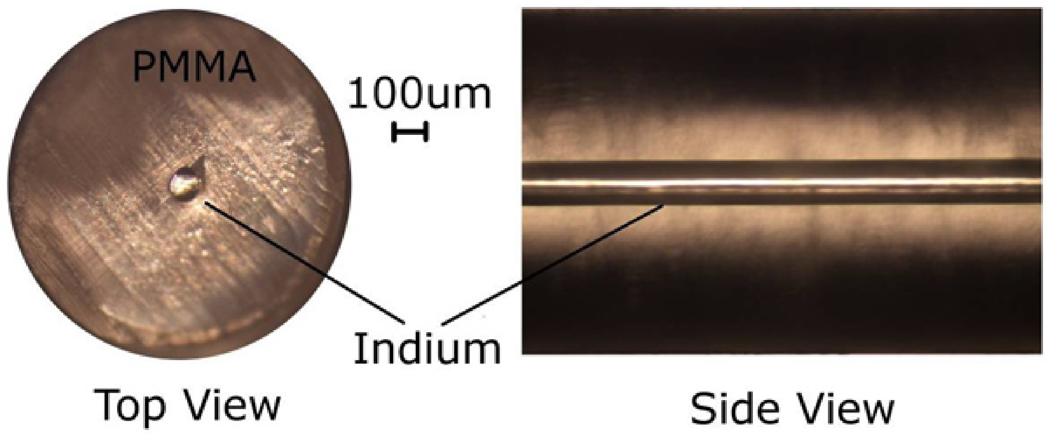

Figure 2. Top view and side view of 1 mm PMMA fibre with a single 100 µm indium wire.

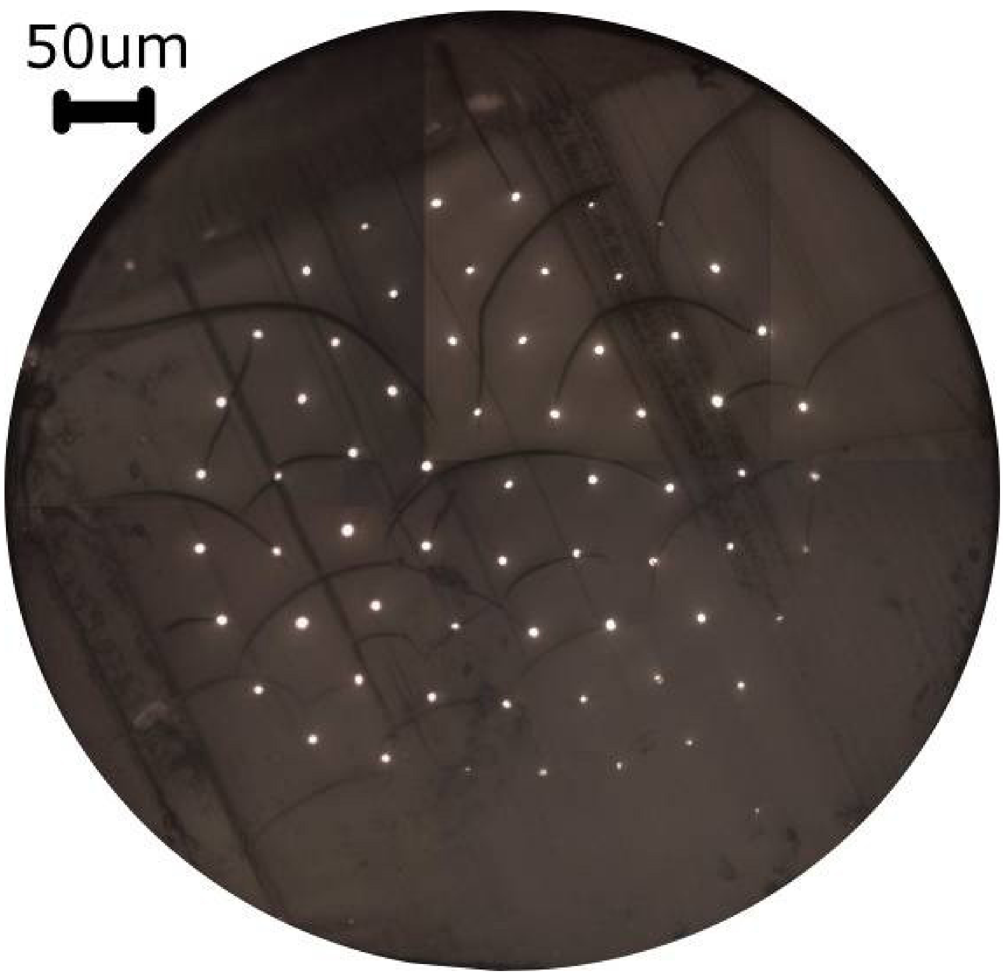

Figure 3. (Composite) optical microscope cross sectional image of the 5 µm indium wires separated by 50 µm in a PMMA fiber. (40x objective lens).

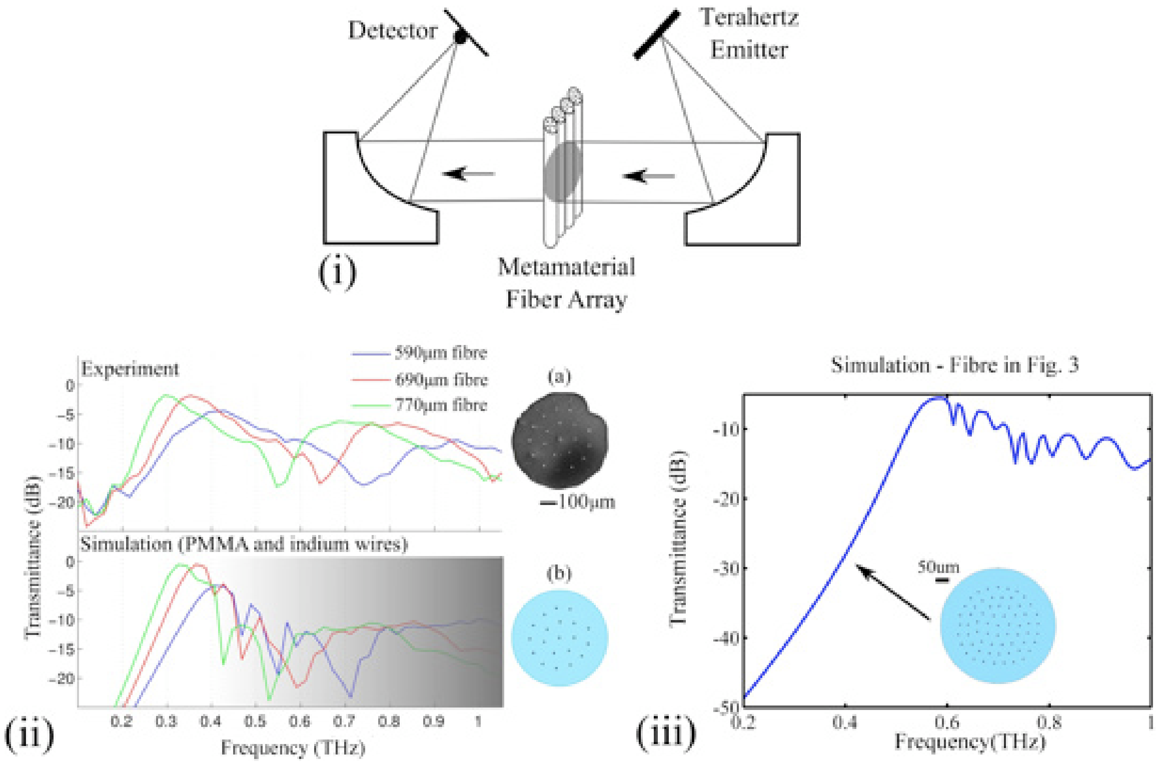

Figure 4. (i) Schematic of the experimental setup for measuring metamaterial fiber transmittance. (ii) (a) Experimental and (b) simulated (finite element method) transmittance for arrays of metamaterial fibers of different diameters (electric field parallel to the wires), as presented in Ref. [8], showing very good agreement. A scanning electron microscope image of the 590 µm fiber is shown in the inset of (a). An image of the simulated geometry is shown in the inset of (b). The smallest fiber had ~8 µm diameter wires separated by ~100 µm. The shaded region illustrates where the medium cannot be seen as homogeneous. The plasmonic transition region shifts to lower frequencies as we increase the fiber diameter (obtained simply by changing the draw speed), resulting in a shifting of the high-pass filtering behavior. After Ref. [8]. (iii) Simulated transmittance for an array of the metamaterial fiber shown in **Figure 3**, using the same methods and optical parameters presented in Ref. [8]. Note that in this case the fiber would exhibit a plasma frequency around 0.6 THz. Click here to view larger figure.

Figure 5. Top section of the fibre draw tower on the secondary side. Note in particular the chuck feed (top) and the furnace (middle), connected to the control unit (right).

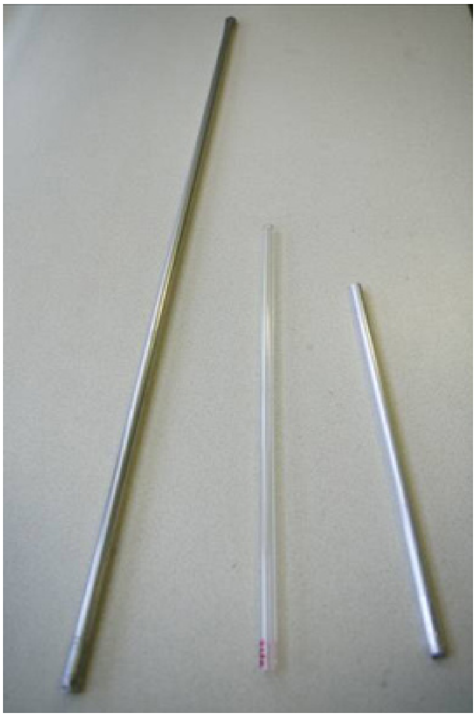

Figure 6. (Left to right) Bottom extender, preform, and top extender.

Figure 7. Attaching top extender - with PTFE (left) and reflective tape (right).

Figure 8. Attaching bottom extender - reflective tape.

Figure 9. Hot air gun crimp.

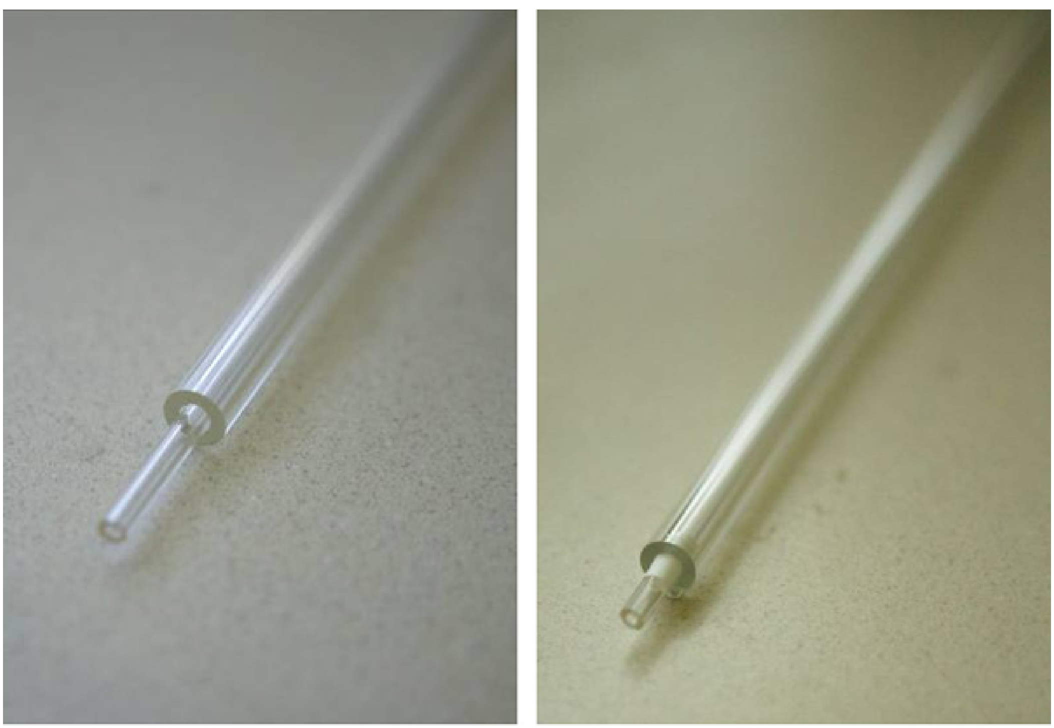

Figure 10. Inserting tube in jacket (left) and with PTFE seal (right).

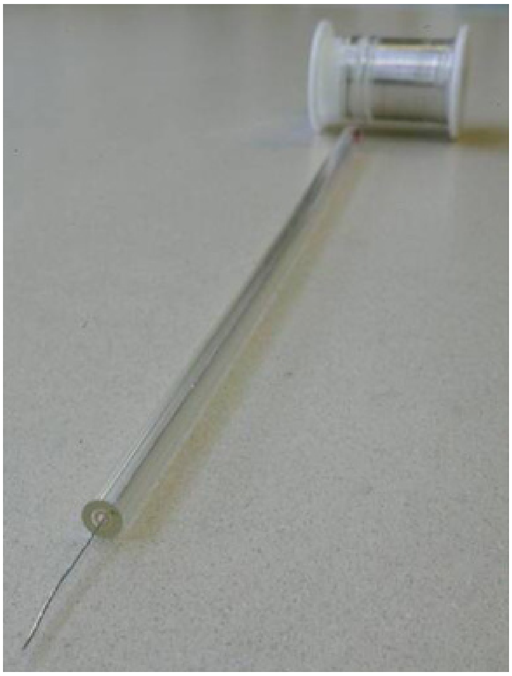
Figure 11. Inserting indium wire into PMMA tube.


Figure 12. Inserting indium wire stacked bundle into PMMA tube.

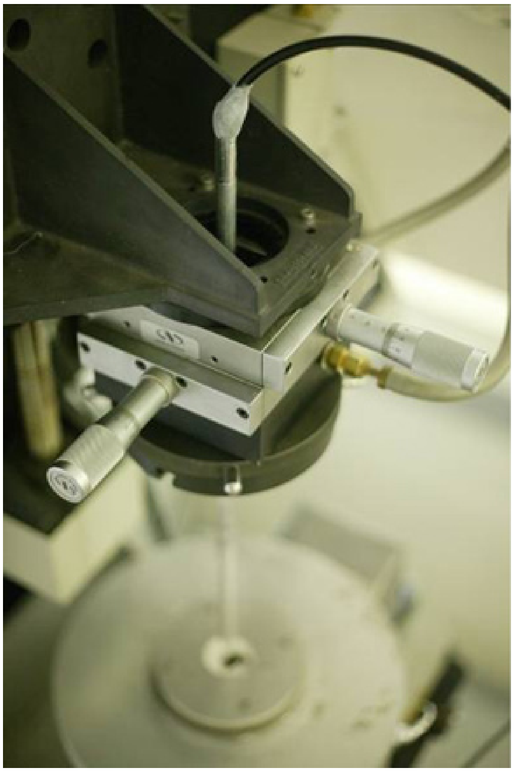

Figure 13. Top to bottom: attaching vacuum tube to preform, clamping preform in the 3 jaw chuck feed unit, and feeding into the furnace.

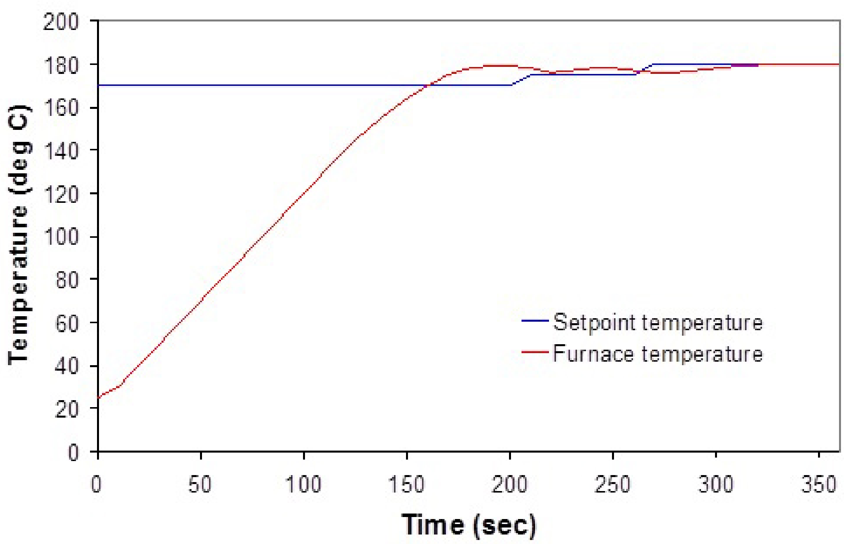

Figure 14. Pre-heat profile.

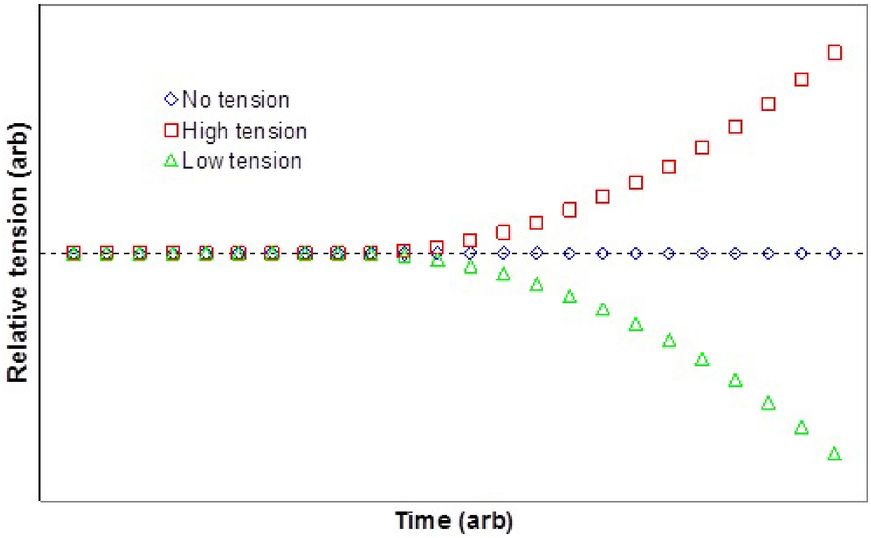

Figure 15. Primary tension profile.

Figure 16. + Drop-down preform section.

Preform OD (mm)	Feed Rate (mm/min)	Draw Rate (mm/min)	Furnace Temperature (°C)
12	2.5-5	25-50	185-200
12	5-10	15-25	185-200
12	10-15	10-20	185-200

Table 1. Primary draw conditions.

Preform OD (mm)	Feed Rate (mm/min)	Furnace Temperature (°C)	Draw Tension (g)
12	10	220-240	70-80
12	7.5-10	210-230	70-80
12	5-7.5	200-220	70-80

12	2.5-5	190-210	70-80
12	1-2.5	180-200	70-80

Table 2. Secondary draw conditions.

Discussion

The technique presented here allows the fabrication of kilometers of continuous three-dimensional metamaterials with microscale feature sizes, possessing a plasmonic response (and thus a tailored electric permittivity) in the THz range, effectively behaving as a high-pass filter. This can be experimentally characterized using terahertz time-domain spectroscopy [11]. Such fiber-shaped metamaterials can be cut and stacked into bulk materials to realize a large number of devices, or woven into other structures, for example negative refractive index materials, when combined with metamaterial fibers possessing a negative magnetic permeability in this range [12]. Note that magnetically responsive fibers may also be fabricated in bulk by a variation on the technique presented here [13].

Disclosures

No conflicts of interest declared.

Acknowledgements

This research was supported under Australian Research Council's Discovery Projects funding scheme (project number DP120103942). B. T. K. and A. A. are the recipients of an Australian Research Council Future Fellowship (FT0991895) and Australian Research Fellowship (DP1093789) respectively.

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