

FABRICATION AND CHARACTERIZATION OF NEGATIVE CURVATURE HOLLOW CORE POLYMER OPTICAL FIBERS FOR NEAR-INFRARED LIGHT GUIDANCE

A THESIS SUBMITTED TO
THE GRADUATE SCHOOL OF ENGINEERING AND SCIENCE
OF BILKENT UNIVERSITY
IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR
THE DEGREE OF
MASTER OF SCIENCE
IN
MATERIALS SCIENCE AND NANOTECHNOLOGY

By
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July 2023

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We certify that we have read this thesis and that in our opinion it is fully adequate,
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ABSTRACT

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M.S. in Materials Science and Nanotechnology

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July 2023

Polymer optical fibers (POFs) have attracted significant attention for their short-distance data transmission, industrial automation, and chemical and biological sensing applications. The low cost, lightweight, flexibility, accessibility, and ease of material processing features of the polymers make them superior to their silica counterparts. Moreover, compared to conventional POFs, hollow core polymer optical fibers (HCPOFs) exhibit light guidance through the air, significantly reducing material absorption loss in the near-infrared (NIR) region. Structuring the cladding part with the appropriate fiber material can further modify the light-guiding properties of HCPOF with low transmission loss in NIR. Several methods have already been employed for the successful fabrication of POFs, but the possibility of fabricating intricate geometry-based HCPOFs with these approaches and optimization of fabrication methods are yet to be resolved.

This study explored the stack and draw technique and fused deposition modeling (3D printing) approaches to find the fabrication feasibility of long-length and intricate geometry-based negative curvature-based HCMPOFs with two different polymeric materials. A detailed investigation was carried out on the modified thermal drawing process to achieve well-structured HCMPOFs directly drawn at high tension from the fabricated preforms. Moreover, during the thermal drawing, expansion and contraction of the core and cladding part of the fibers were frequently observed. Inflation of the cladding tubes during the fiber drawing was required to preserve the designed structure in the fibers. This was achieved by applying gas pressurization inside the fibers in both preforms made by the stack and draw technique and 3D printing.

Optical characterization is performed using Supercontinuum (SC) Laser in the 600 – 1700 nm wavelength range. Differences in the transmission spectra between core and cladding structures significantly prove the light-guiding properties of the proposed HCMPOFs. The transmission losses of the HCMPOFs were measured using Optical Spectrum Analyzer (OSA), and were found to average 49.26 dB/m for stack and draw-based fabricated six-tube HCMPOF and 16 dB/m for 3D printed six-pointed star cladding-based HCMPOF. Further investigation is carried out on bend-induced loss against the mechanical effects of the 3D printed intricate geometry-based HCMPOF at different bending angles. The lower transmission loss with a low bend-induced loss against the mechanical effects of HCMPOFs explicitly shows the potential of using HCMPOFs as an alternative to conventional polymer optical fibers for visible and infrared light guidance.

Keywords: Hollow-core negative curvature polymer optical fibers (NC-HCPOFs), Near-infrared (NIR), Stack and draw technique, Fused deposition modeling, Polycarbonate, Transmission loss, Bend loss.

ÖZET

YAKIN KIZILÖTES İİK YÖNLENDİRMESİ ÇİN DİBÜKEY HOLLOW CORE POLİMER OPTİK FİBERLERİN ÜRETİMİ VE KARAKTERİZASYONU

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Temmuz 2023

Polimer optik fiberler (POF), kısa mesafeli veri iletimi, endüstriyel otomasyon, kimyasal ve biyolojik algılama uygulamaları için büyük ilgi görmüştür. Polimerlerin düşük maliyeti, hafifliği, esnekliği, erişilebilirliği ve malzeme işleme kolaylığı özellikleri onları silika muadillerinden üstün kılar. Ayrıca, geleneksel POF'lar ile karşılaştırıldığında, hollow core polimer optik fiberler (HCPOF), yakın kızılötesi (Near Infrared, NIR) bölgesindeki malzeme absorpsiyon kaybını önemli ölçüde azaltan hava yoluyla ışık yönlendirmesi sergiler. Kaplama bölümünün uygun fiber malzeme ile yapılandırılması, NIR'da düşük iletim kaybı ile HCPOF'un ışık yönlendirme özelliklerini daha da değiştirebilir. POF'ların başarılı bir şekilde üretilmesi için çeşitli yöntemler halihazırda kullanılmıştır, ancak bu yaklaşımlarla karmaşık geometrili HCPOF'lar üretme olasılığı ve üretim yöntemlerinin optimizasyonu henüz çözümlenememiştir.

Bu çalışmada, iki farklı polimerik malzeme ile karmaşık geometrili dışbükey HCPOF preformlarının fabrikasyon uygunluğunu bulmak için stack and draw tekniği ve fused deposition modelleme (3D baskı) yaklaşımları araştırıldı. Üretilen preformlardan doğrudan yüksek gerilimde çekilen iyi yapılandırılmış HCPOF'lar elde etmek için modifiye edilmiş termal çekme işlemi üzerinde ayrıntılı bir araştırma yapıldı. Ayrıca, ısı çekme işlemi sırasında, fiberlerin çekirdek ve kılıf kısımlarında genleşme ve büzülme sıklıkla gözlenmiştir. Fiberlerdeki tasarlanan yapıyı korumak için kaplama tüplerinin fiber çekme işlemi sırasında şişirilmesi gerekmiştir. Bu, stack and draw tekniği ve 3D baskı ile yapılan preformların her ikisinde de fiberlerin içine gaz basıncı uygulanmasıyla sağlandı.

Optik karakterizasyon, 600 – 1700 nm dalga boyu aralığında süper süreklilik

ışık kaynağı kullanılarak gerçekleştirilmiştir. Çekirdek ve kaplama yapıları arasındaki iletim spektrumlarının farkı, önerilen HCPOF'larının ışık yönlendirme özelliklerini önemli ölçüde kanıtlamaktadır. HCPOF'ların iletim kayıpları, optik spektrum analiz cihazı kullanılarak ölçüldü ve stack and draw tekniği kullanılarak üretilen altı tüplü HCPOF için ortalama 49.26 dB/m ve 3D baskılı altı köşeli yıldız kaplama kullanılan HCPOF için 16 dB/m olarak bulundu. İlâveten, 3D baskılı karmaşık geometrili HCPOF'ların mekanik etkilerine karşı bükülme kaynaklı kayıp (bend-induced loss) üzerinde farklı bükülme açılarında araştırma yapılmıştır. HCPOF'ların mekanik etkilerine karşı düşük bükülme kaynaklı kayıp ile elde edilen daha düşük iletim kaybı, görünür ve kızılötesi ışık kılavuzluğu için geleneksel polimer optik fiberlere bir alternatif olarak HCPOF'ların kullanılma potansiyelini açıkça göstermektedir.

Anahtar sözcükler: Dışbükey Hollow-core Optik Fiberler (NC-HCOF), Yakın İnfrared (NIR), Stack and Draw Tekniği, Fused Deposition Modellemesi, Polikarbonat, İletim Kaybı, Eğilme Kaybı.

Acknowledgement

I acknowledge that First and foremost, I want to express my deepest gratitude towards my supervisor, Dr. Mustafa Ordu, for his unwavering encouragement, guidance, and support during my Master's studies. I am grateful to him for having faith in me, taking me into Contemporary Fibers Laboratory (CFL), and allowing me to improve my skills under his supervision. Coming from a different background, coping with another advanced research field, such as fiber optics, was initially difficult. Whenever we faced scientific problems, his sincere guidance and precious feedback helped me through it, without which reaching this position would be more challenging. I learned many things in the fiber optics research field from him. I also discovered him to be someone from whom I could learn how to work in a team with people from diverse backgrounds and specialties while keeping a healthy balance between work and social life.

I want to express my gratitude towards my thesis committee: Asst. Prof. Abdullah Demir, Assoc. Prof. Engin Durgun and Prof. Evren Mutlugün for their constructive comments and feedback. I also would like to thank Asst. Prof. Bülend Ortaç, along with his group members (Dr. Ali Karatutlu, Dr. Esra Kendir, Elif Yapar Yıldırım, and Seyitali Yaşar) for their guidance, support, and help during the optical characterization in my research work. By providing the equipment, direction, and assistance required for fiber manufacturing and optical measurement, they have sincerely helped me throughout this research work. Thanks to engineer Mr. Can Guven for his assistance in the 3D printing process and other things related to this research work. I want to thank UNAM, Bilkent University, and TUBTAK for helping me to complete my Master's research work by providing an excellent research atmosphere and financial support. I also thank Asst. Prof. Talip Serkan Kasırga for allowing me to start my M.Sc. at UNAM, Bilkent University. I sincerely thank all of my fellow CFL group members (Abdul Aziz Khan, Ali Bagheri Behboud, Asfandiyar Khan, Arda Kurucu, Md Kawsar Ahmed, Md Mehdi Hasan, Md Sazid Bin Sadeque, Muhammad Zain Siddiqui, Cihat Kürşat Kalkan, Alper Koçak, Berke Ozyalın, Ceren Dilsiz, Deniz Berk Onuralp, Ilgın Albasar, Göktan Yılmaz, Manahil Noor, and Murat Barış) for

their helping hands whenever needed as well as fantastic time we had together. I especially would like to thank Berke Ozyahn, Ceren Dilsiz, and Md Sazid Bin Sadeque for being a supportive partner in this project. I also want to thank my friends from the Afghan, American, Arab, Bangladeshi, Indian, Iranian, Pakistani, and Turkish communities here at Bilkent University. They always believed in me, supported me, and were there for me whenever I needed them.

Above all, I would like to thank my dear parents, Md. Abdur Razzak, Shahanaaj Najnin, and my most beloved sibling, Ehan Sadat Shorup, my grandparents and aunties, for their continuous support. Especially my brother, who was always there whenever I needed him. Whatever I have achieved so far would be impossible without them. I thank you all for everything. Finally, I would like to show my gratitude to the Father of the nation of my country Bangabandhu Sheikh Mujibur Rahman, and those freedom fighters like my grandfather Md Moksedur Rahman Bablu, who sacrificed everything during the Liberation War of Bangladesh in 1971. I firmly believe that without their sacrifice, I would never be able to come to this stage of my life.

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Chapter 1

Introduction

1.1 Hollow Core Fibers

Optical fibers have great significance in long-length telecommunication and other essential applications, such as high-power laser delivery and biological and chemical sensing [17, 18, 19, 20]. Conventional solid core optical fibers (SCOFs) made of silica or polymers guide light based on the total internal reflection (TIR) mechanism. In SCOFs, the core is made of higher refractive index (RI) material and is surrounded by a lower RI-based cladding structure. Even though these types of fibers propagate light as an excellent waveguide, they have certain limitations, especially in the ultraviolet (UV), visible, and near-infrared (NIR) wavelength ranges, where Rayleigh scattering is dominant in silica and polymer materials. This Rayleigh scattering loss restricts the minimum theoretical optical propagation loss up to a specific limit, such as conventional SCOF 0.15 dB/km [1].

By contrast, the invention of hollow core fibers (HCFs) proved to be a better optical waveguide by overcoming those limitations. In HCFs, light propagates through the air core surrounded by a higher RI-based cladding structure. So, the index guidance mechanism doesn't work out in these fibers. Yet, HCFs have better optical properties, such as low dispersion, a higher optical damage threshold,

and low latency [21, 22, 23, 24, 25, 26, 27, 28, 29, 30, 31, 32, 33, 34, 35, 36, 37, 38, 39, 40, 41]. Hence, these properties make HCF suitable for different applications than their SCOF counterparts.

Simple capillaries are one of the examples to be used as HCFs. These fibers with higher RI cladding material (silica or polymers) surrounded by the air core have a more straightforward fabrication process. Yet, they come with higher transmission losses [23, 42]. This limitation makes them unsuitable for long-range telecommunication applications. Even though a larger core-based HCF can reduce transmission loss in some contexts, it will also reduce the light intensity guided through the core. Low transmission loss with better modal and bending properties against mechanical effects is desirable for specific applications. So, these HCFs are not eligible for high-power laser delivery and single-mode applications [37, 38, 40].

After several years of efforts in this research field, the discovery of low-loss HCF was achieved by structuring the cladding geometry. The fundamental light-guiding mechanisms of these successfully fabricated HCFs are photonic bandgap (PBG) and anti-resonant optical waveguide (ARROW) models or inhibited coupling (IC) theory. These HCFs include PBG fibers, kagome HCFs, and negative curvature HCFs.

1.2 Types of Hollow Core Fibers

The light guidance mechanism for HCFs is complicated and needs to be better understood. However, HCFs are generally classified into two categories based on their light guidance properties: PBG and negative curvature HCFs (NC-HCFs). Figure 1.1 provides a brief evolution of the development of distinct types of low-loss HCFs.

PBGFs are the first type of HCF with excellent light confinement in the hollow core region. These fibers work on photonic bandgap effects, where periodical

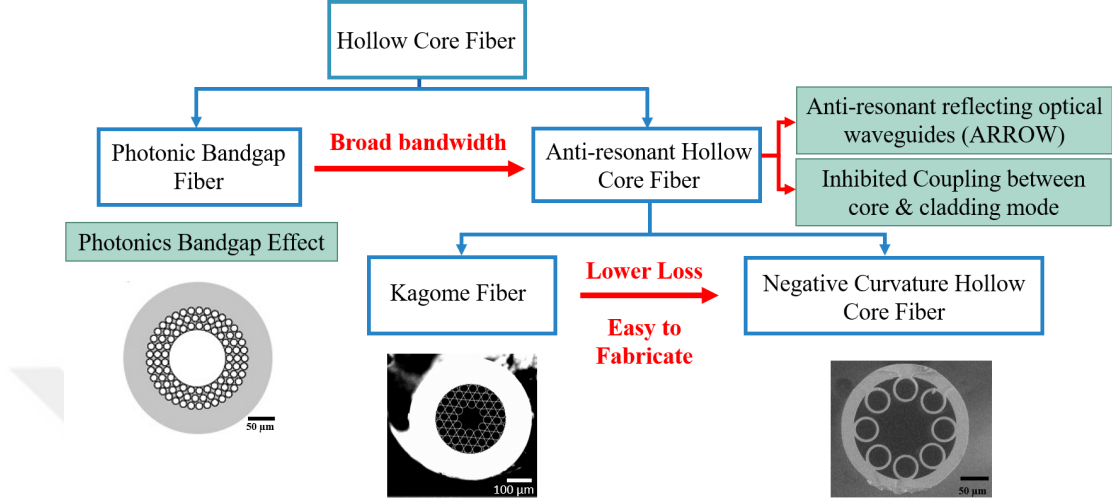


Figure 1.1: Schematic showing distinct types of hollow core fibers with their light guiding mechanisms.

structure creates refractive index (RI) variation in the cladding. Thus, specific wavelengths are allowed to propagate through the core of the fibers [21, 43]. After the first introduction of this concept by Yablonovitch and John et al. [44, 45], it was conceived by Russell et al. in 1991 [21], and Cregan et al. fabricated the first PBG-based HCF in 1999 [44]. Although this type of fiber proved to have low transmission loss (1.2 dB/km at 1620 nm), it suffers from SSL, which indeed restricts further reduction of propagation loss due to its thermodynamic origin [46]. Moreover, the intersection points of the membranes surrounding the core create surface modes and further narrow down the bandwidth. Figure 1.2 (b) provides an SEM image of a fabricated PBGF [47].

The Kagome HCF, which consists of an arrangement of membranes called the Kagome lattice, is the first demonstration of IC HCF with a broader bandwidth, as shown in Figure 1.2 (a) [29, 48, 49, 50]. Light is well confined in these fibers by the strongly inhibited coupling between the core and cladding modes [25]. A significant mismatch in the effective index helps to prevent overlap between those modes and thus confine the light with a wider bandwidth than PBGFs [29, 48, 51, 52, 53, 54]. Moreover, kagome HCF's low loss and tunable dispersion properties present it as a potential candidate for nonlinear optics [48]. Yet, the creation of nodes at the thicker struts between the co-joints of thinner membranes

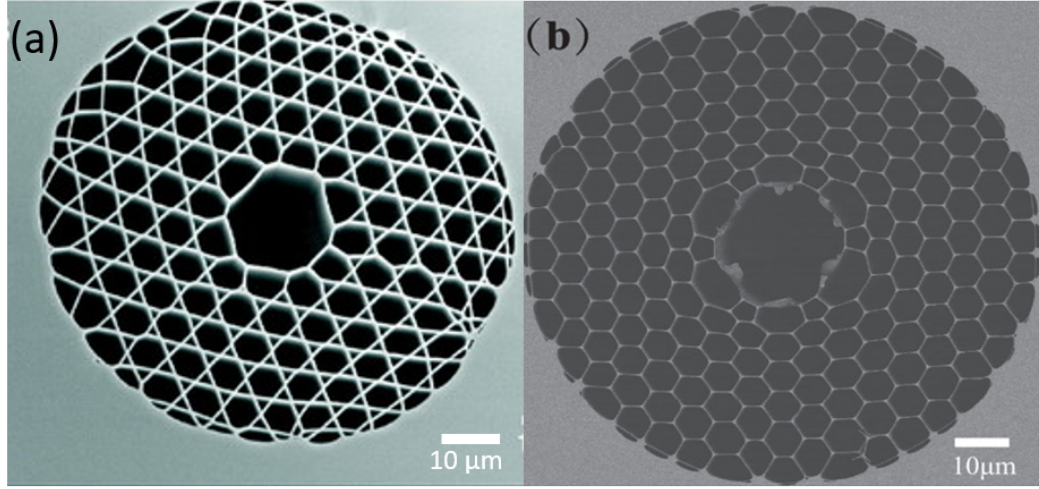


Figure 1.2: Scanning electron microscope images of (a) kagome fiber [33] and (b) PBGF [32].

and fabrication feasibility restrain having a smooth, broad bandwidth and thus necessitate solving. However, kagome HCF was further developed to achieve lower loss by introducing negative curvature surrounding the core interface [55]. These findings were essential for the development of NC-HCFs. Properly structuring the cladding geometry in NC-HCFs reduces the loss further than in Kagome HCFs. At the same time, NC-HCFs possess smooth bandwidth with an easier fabrication process.

1.3 Negative Curvature Nodeless Hollow Core Fiber

The introduction of negative curvature in HCFs came up with promising anti-resonant HCFs to prevent maximum light leakage. The negative curvature fibers (NCFs) can be defined as the radial unit vector of the center of the core in the opposite direction to the surface normal vector of the core boundary [56]. Figure 1.3 represents the mode profile inside three distinct types of curvature (positive, flat, and negative) that can exist in HCFs.

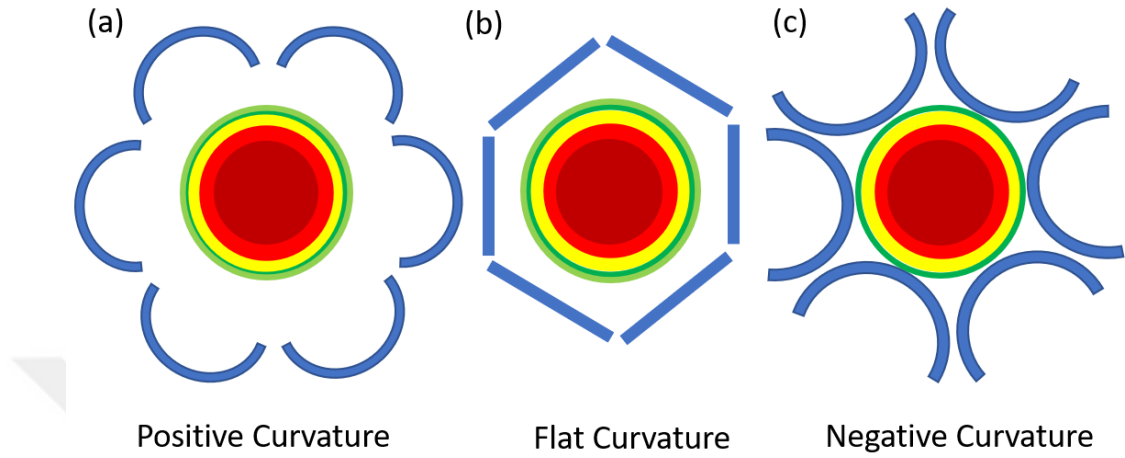


Figure 1.3: Schematics of positive, flat, and negative curvatures between a core-cladding interface in HCFs with their mode profile.

Optical fiber possesses three distinct transmission loss types, as shown in Figure 1.4. However, by extracting the material used as a solid core, material loss (ML) can be reduced. Even though hollow core PBGFs (HC-PBGF) show lower transmission loss, they suffer from SSL. Whereas NCFs have issues with confinement loss (CL) [1, 25]. Structuring the cladding geometry in an engineered way, this CL can be reduced further, for example, Nested Anti-resonant Nodeless Fiber (NANF) design [1, 57, 58].

Even the total transmission loss can be reduced lower than in PBGFs and conventional SCOFs as developed by F. Benabid et al. in 2021 and Poletti et al. in 2014 [1, 2] (shown in Figure 1.5). The unconnected cladding tubes surrounding the hollow core with connected hybrid lattice possess lower transmission loss

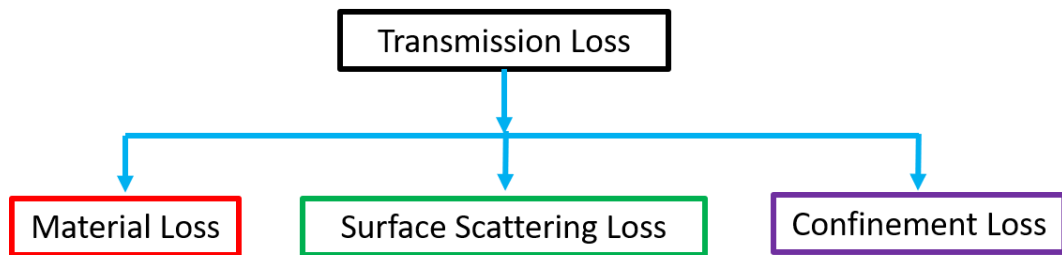


Figure 1.4: Illustration of three distinct reasons (material loss, surface scattering loss, confinement loss) for transmission loss in optical fiber.

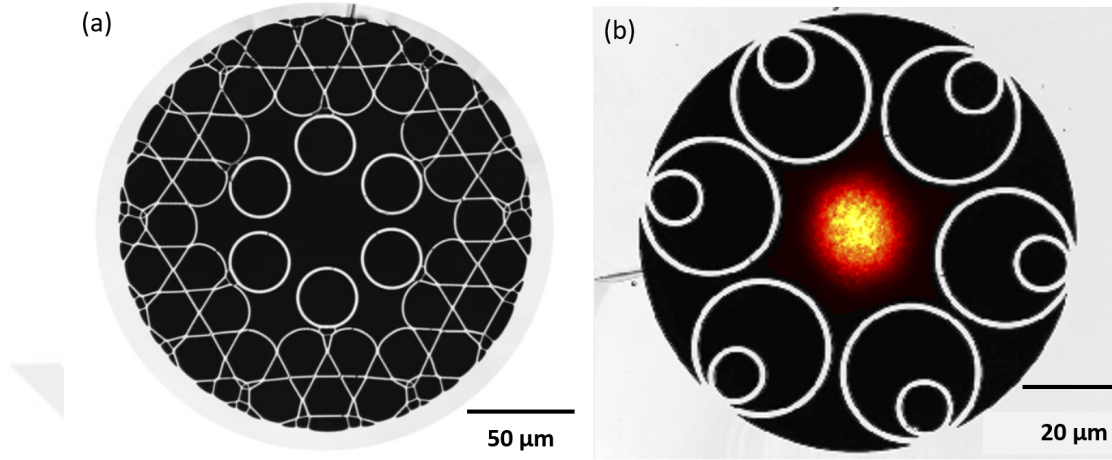


Figure 1.5: Negative curvature HCF with (a) hybrid-lattice and (b) nested cladding (NANF) design to reduce the confinement loss further. [1, 2]

with a smooth broadband transmission window. Further demonstration of structuring the cladding geometry to reduce transmission loss at telecommunication wavelength as well as in mid-infrared (mid-IR) has been demonstrated, for example, six-pointed star cladding tubes and Elliptical Nested Tubular Fiber (ENTF) [57, 58]. However, these complicated cladding designs come with challenges in fabrication feasibility.

1.4 Physical Features of Polymer

The merits of polymers are implied in their physical characteristics. Except for fluorinated polymers, which have higher densities, most polymeric materials possess light weight, flexibility, and better sensitivity to temperature than their silica counterparts. Along with these intrinsic properties, optical fibers made off polymers are preferred because of their low cost, accessibility, and accessible material processing methods. Optical fibers with smaller core diameters for single-mode operation require precise alignment, while large core diameter-based silica optical fibers have a high loss due to tight bending. In contrast, the flexibility property of polymers allows POFs, even with a larger core (1 mm), to be used with better tolerance [59, 60]. Other advantages, such as the light guidance ability in the

visible range, being easily disposable, and not producing shards at the breakage, are also crucial for different applications. These qualities are the main reasons for using POFs for domestic, industrial, and medical use (especially by those with little or no knowledge about optical fiber usage). Furthermore, due to their high drawing temperature, silica optical fibers are incompatible with incorporating organic materials. In contrast, various polymer materials are developed and can be used to fabricate POFs for different applications, such as non-linear optics, biomolecule detection, and emission devices. Organic materials can be incorporated in POFs as they have the same approach on a technological platform, which emphasizes easy material processing, cheaper cost, and similar types of materials. As the surface of some polymers can easily be functionalized with different organic materials, they give an advantage in biomedical applications compared to the other optical fiber counterparts [61, 62, 63].

1.5 Applications of POF

1.5.1 Illumination

POF has been inherited as one of the largest applications for illumination purposes. POF typically has a big core, high light-collection ability, and flexibility for illumination optics. Traditionally POF has been employed as a point source for most illumination applications, such as highly focused spotlights in confined spaces or structures [64]. However, doping the polymers with scattering particles has increased its ever-growing demand as side-scattering POF. This type of POF can be used up to several meters for strip lighting when added with a light source.

1.5.2 Sensing

After illumination optics, most of the POFs are used for various sensors. As a strain sensor POFs have been used to detect crack propagation in concrete beams

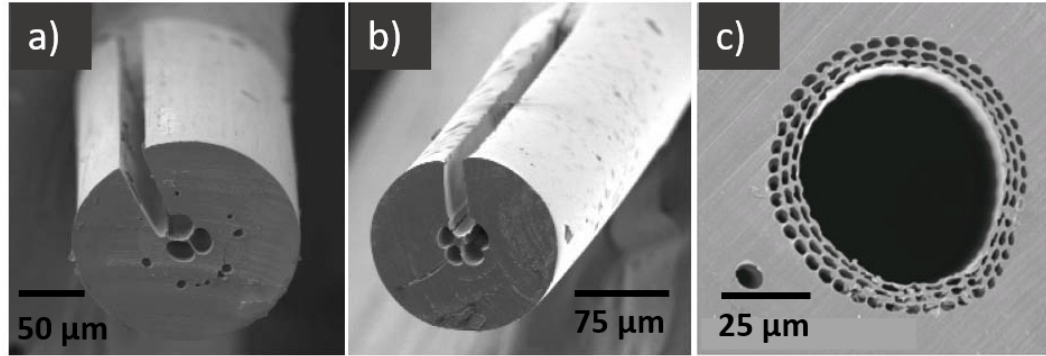


Figure 1.6: a) 3-hole and b) 5-hole design slotted cladding-based fiber (140 μm) as pH sensor [3], c) HCMPOF fiber used as chiral fiber and potential gas sensor [4].

[65], damage assessment for composite materials [66], and as a humidity sensor [67]. These prototypes are shifting from laboratory experiments to the commercial market, especially for the industries as primary customers. Reviews on the potentiality of POF sensors provide detailed information about modifying fiber improvements [68]. The appearance of multimode MPOF has brought more significance to the potential application of sensors. As the micro-structured holes in MPOFs can contain solutions compared to solid core POFs, these fibers can effectively be used for chemical or gas sensing via changing properties. For example, modifying the surface structure of the TOPAS-based MPOF has been used as an antibody sensor. The fiber was coated with the receptor of a specific antibody, and the presence of it was detected using the concept of capturing selective antibodies [48]. Similarly, a pH range of 2.5–4.5 sensitivity has been demonstrated by coating the micro-structured holes of an MPOF using pH-sensitive fluorescence dye-doped (eosin) cellulose acetate (CA) thin-film [62].

Opening up the mPOF through its lateral axis for a long length can also be an excellent solution for a pH sensor, as demonstrated using a bromothymol blue (BTB) indicator for the sensing characteristics test. This demonstration showed that a laterally opened fiber immersed in water mixed with BTB indicator significantly compared basic and acidic solution with its different transmission spectra from 500 nm to 800 nm wavelength range [3]. Moreover, hollow core MPOF (HCMPOF) successfully showed the possibility of chemical sensing using

the band gap shifting mechanism inside the hollow core. As a proof of concept, the fiber was filled with water and showed different transmission peaks than the air. Use as chiral fiber was also demonstrated with the optical rotation of this fiber [4].

1.5.3 High-Speed Data Transmission

There is no alternative to POF for growing demand regarding high-speed short-distance data transmission for home, industrial, and automotive appliances, consumer electronics, and local area networks (LAN). Easier handling processes and manufacturing costs for mass production with flexibility of usage play a vital role in choosing suitable optical fiber. Until now, step-index POF possessed significant reliability for high-speed data transmission over 50 m with a 400 MB/s rate for consumer electronics. In the automotive industry, also known as 'killer application,' the importance of POF is expected to grow to US\$ 567 M [69].

However, the inadequacy of step index fiber in increasing bit rate, graded index (GI) fibers are a better alternative with improved bandwidth. Despite having optical fibers in the street, very few people possess them in their homes and industry. This simple fact has come with the significance of the GI-POF in different applications. Installation of POFs with high-speed data rates has earned significant importance for telephone, internet, and television connections while uprooting the copper-based systems in terms of cost-saving procedures. Even though silica fibers are still working as a backbone within a building with wireless or copper cable-based connections to the consumer end, fluorinated POF, as well as PMMA-based POF, can also be used as an alternative for high-speed data transmission with large bandwidth in the infrared and visible regimes over several meters at a lower cost.

Moreover, concerning durability and flexibility, POFs with a large core are better than their silica fibers counterparts. Considering the manufacturing costs, mass production, and data transmission, the MPOFs' reliability still needs to be tested despite their huge advantages over step-index POF. But soon, with

suitable fabrication methods with mass manufacturing facilities, HCMPOF can surpass any of the optical fibers mentioned above for short-distance high-speed data transmission at a lower cost.

1.6 Historical Progress on HCMPOF

1.6.1 HCMPOF based on Stack and Draw Technique

Figure 1.7 illustrates the historical progress on fabricated HCMPOFs based on stack and draw technique. A. Argyros et al. first introduced the stack and draw technique for fabricating HCMPOF [5]. This polycarbonate (PC) material-based hollow core POF consisted of two layers of microstructured capillaries surrounding the air core. The 400 μm outer diameter-based HCMPOF had a core of 61 μm . Two strong transmission windows were observed at visible and NIR regimes. The propagation loss was 9 dB/m at 800 nm and 3.1 dB/m in the 1550 nm wavelength regimes. The other two images (negative curvature POFs and lotus fiber) represent our work.

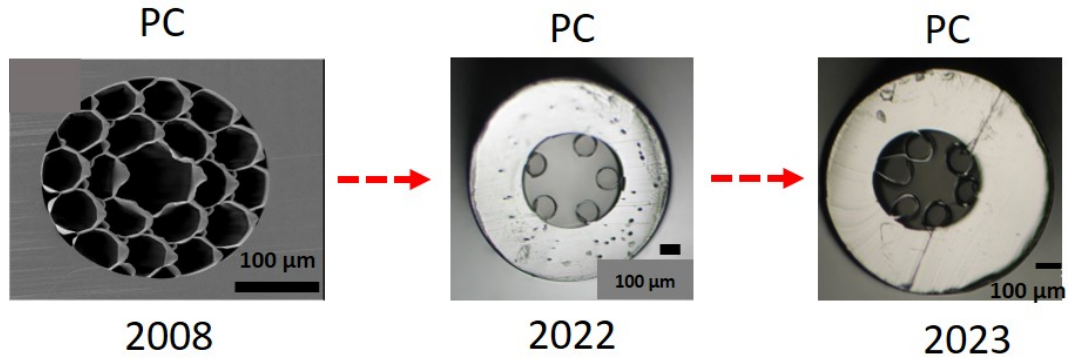


Figure 1.7: An illustration of historical overview of stack and draw based preform to fiber fabrication of kagome structured [5], five tubes, and six tubes based tubular HCMPOF.

1.6.2 3D Printed HCMPOF

A historical progress on recently developed and fabricated POFs from 3D printed preforms is summarized in Figure 1.8, whereas Table 1.1 contains a brief overview regarding fabricated different types of fibers from various 3D printing filament materials and their optical performances.

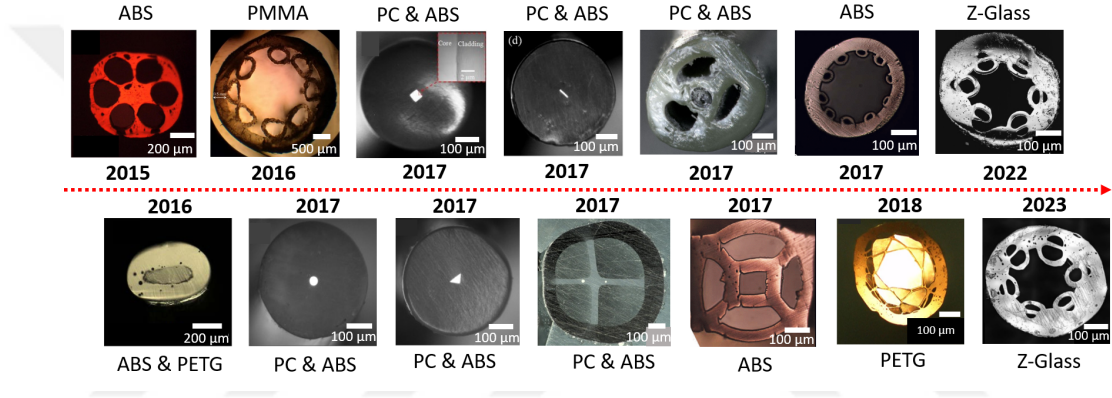


Figure 1.8: An illustration of historical overview of 3D printed preform to fiber fabrication [6, 7, 8, 9, 10, 11, 12, 13].

As proof of concept, the first 3D printed preform to air structured SCPOF fabricated by K. Cook et al. demonstrated 0.75 dB/cm propagation loss at 1064 nm. The 3D printing filament (ABS material) based fiber required no gas pressure while the thermal drawing process [6]. The air-structured SCPOF also showed promising light-guiding properties at visible (633 nm) and near-IR (1550 nm) wavelengths. Yet, the geometry of the fiber following the thermal drawing process possessed a slight deformation owing to a lack of proper polymerization and the formation of air bubbles during 3D printing. A similar phenomenon was observed in step-index SCPOF and special core-shaped fibers fabricated by K. Cook et al. and Qikai Zhao et al., respectively [7, 8].

These fibers showed better light guidance in the visible wavelength (0.42 - 1.8 dB/cm). Since the structures are solid core based, they didn't possess serious deformations in the fiber geometry. On the other hand, 3D-printed hollow core POFs (HCPOFs) sustained severe damage in the fiber geometry during the fabrication process, as presented in [9, 10, 11, 12].

Table 1.1: A comparison of the recently fabricated POFs from 3D printed preform [6, 7, 8, 9, 10, 11, 12, 13]

Year /Ref	Preform type	Core material	Cladding material	Fiber Dia (μm)	Propagation Loss (dB/cm)
2015 [60]	Air structured	ABS	ABS	600-700	0.75 (1064 nm)
2016 [61]	Step index	ABS	PETG	200-300	0.42 (1052 nm)
2016 [66]	Hollow core eight tube cladding	Air	PMMA	4700 (cane)	No measurement
2017 [62]	Circular shaped core	PC	ABS	500	0.7 (633 nm)
2017 [62]	Square shaped core	PC	ABS	500	1.2 (633 nm)
2017 [62]	Triangular shaped core	PC	ABS	500	1.3 (633 nm)
2017 [62]	Rectangular shaped core	PC	ABS	500	1.8 (633 nm)
2017 [63]	Microstructured monofilament	PC (extruded)	PC (printed)	520-620	No measurement
2017 [63]	Microstructured monofilament	Air	Green ABS	520-620	No measurement
2017 [65]	Rectangular hollow core	Air	ABS	250	Guidance demonstration
2017 [65]	Circular hollow core	Air	ABS	170	No measurement
2018 [64]	Hollow core with periodic cladding	Air	PETG	466	0.2 (4000 nm)
2022 Our work [67]	Hollow core with intricate geometry	Air	Z-Glass	430	0.325 (1300 nm)

At first, the thermal drawing of HCPOFs already poses certain challenges due to the requirement of extra attention to keep the capillaries in a structured way. Again, the non-Newtonian fluidic behavior of 3D printing polymer filaments mixed with different additives adds another issue [70]. Furthermore, Several efforts were also put into pre and post-annealing processes of the 3D printed preform to achieve maximum transparency [71]. Among several efforts to fabricate HCPOFs, W. Talataisong et al. demonstrated better loss (0.2 dB/cm) at 4 μm wavelength [64]. Yet, there was no resonance band due to the serious deformation of the fiber geometry. Despite having practical challenges, these fabricated mPOFs utilizing the fused deposition modeling (FDM) approach provided significant scopes for the fabrication of intricate geometry, such as structuring the outer cladding region of hollow-core fibers to explore further the influence of outer cladding designs on the losses [57, 13]. Negative curvature cladding elements in hollow-core fibers have provided important breakthroughs for low-loss guidance [1].

1.7 Objectives

POFs provide better flexibility even at larger core diameters, easier handling process, and cheaper material cost than silica counterparts. Despite these advantages, the successful use of POFs for optical transmission has halted due to several other drawbacks, such as high material absorptions. Propagation loss in POFs is dominated at short wavelengths by Rayleigh Scattering and at longer wavelengths (NIR region) by the polymer structure's harmonic vibration of C-H bonds. Several efforts were investigated in developing polymer materials' properties, yet the propagation loss remained high. Since propagation loss in HCFs is irrespective of material loss, these fibers came hoping to use POFs instead of expensive copper cables for short-distance high-speed data transmission. This thesis focuses on the possible fabrication and characterization of HCMPOFs for potential use in short-distance data transmission purposes. Two different fabrication approaches (stack and draw technique and FDM) are investigated for the possible fabrication of the HCMPOF preform following the thermal drawing process of the fiber.

Light guidance characteristics, such as straight transmission loss, bend induced loss, are evaluated for both HCMPOFs with two approaches. In HCFs, light confinement plays a significant role in reducing the propagation loss. This loss can further be reduced by structuring the outer cladding structure of the geometry [1, 57, 72, 73]. The fabrication possibility of these geometries is explored in this research work with lower propagation loss reported till now. The problems regarding the fabrication of well-structured POFs and their higher transmission loss (especially for stack and draw-based fabricated HCMPOFs) are also explained. By carefully considering solutions to those problems, well-structured geometry with low-loss optical propagation can be successfully achieved.

Chapter 2

Concepts of Guiding Mechanism, Fabrication and Characterization Method

2.1 Light Guiding Mechanism inside Hollow Core

The light guiding mechanism in HCFs is not based on TIR. In contrast, the guidance mechanism is a bit complex and not well understood yet. Several light-guiding mechanisms, such as Marcatili and Schmeltzer, ARROW, IC, and multiple-layer models, have been proposed to explain light-guiding properties inside NC-HCFs.

2.1.1 ARROW Model

Both ARROW and IC theory support leaky modes instead of guiding modes inside the HCFs. According to the Arrow model, the cladding structures consist of high and low RI materials that act together as Fabry-Perot resonators [26, 51, 52]. As the light wave propagates through the hollow core, it creates constructive and destructive interferences at resonant and anti-resonant wavelengths. Light propagates through the hollow core at anti-resonant wavelengths leaving other resonant wavelengths leaked out through the cladding structure of the fiber. Several transmission bands are formed between these resonance peaks and can precisely be calculated with this theory. These resonant peaks are only dependent on the thickness of the cladding structures and can be given as follows:

$$\lambda_m = \frac{2t\sqrt{n_1^2 - n_2^2}}{m} \quad m = 1, 2, 3... \quad (2.1)$$

Where λ_m represents the resonance peaks, n_1 and n_2 are low and high refractive indices of the cladding structures, respectively. t is the thickness of the cladding tubes and m is the positive integer number at different resonant points of $m = 1, 2, 3, 4$.

However, according to the ARROW model, the cladding tubes' thickness is the same [26, 52]. Hence it cannot calculate the resonance peaks, where multiple high and low refractive indices materials with different thicknesses are used. Moreover, this model is not helpful for the exact propagation loss measurement as it has been derived from a simplified physical model. Yet, for precise calculation of the resonance wavelength, this model can be used efficiently [1, 26, 51, 52, 74, 75, 76, 77, 78, 79, 80, 81, 82, 83].

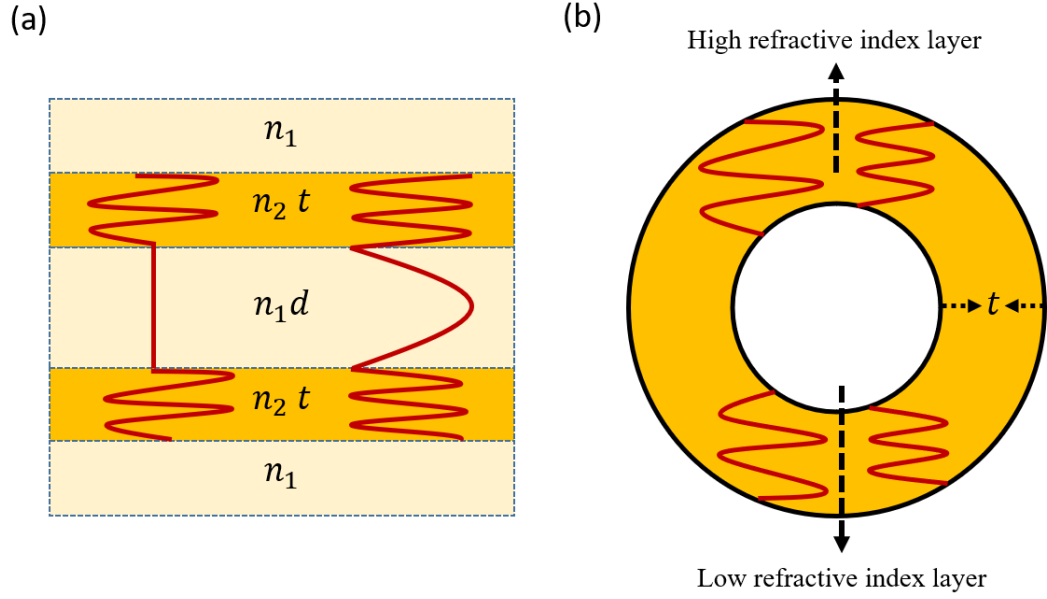


Figure 2.1: Schematic illustration of the ARROW model effect in a (a) simple waveguide and (b) cladding tube of NC-HCFs.

2.1.2 Inhibited Coupling

Figure 2.2 shows several types of modes that coexist in NC-HCFs. The core modes (Fig. 2.2 (a)) refer to the modes guided through the HCFs' core. On the other hand, NC-HCFs also possess cladding modes inside the hollow cladding tubes (Fig. 2.2 (b)). Dielectric tube modes propagate through the cladding materials, as shown in Fig. 2.2 (c).

Strong coupling between these core and cladding modes is the reason for high CL. When the phase match conditions are satisfied, the leaking fundamental core modes are strongly coupled with the existing cladding modes inside the NC-HCFs. Hence, it reduces the effective indices differences between core and cladding, and the transfer or exchange of energy between the core and cladding modes occurs [84]. According to IC theory, minimum or no coupling between these core and cladding modes is possible under particular phase mismatch conditions and low spatial overlap between their electric fields [1, 25, 26, 52, 77, 85]. Hence, CL can be reduced in NC-HCFs. This CL can further be reduced by considering other

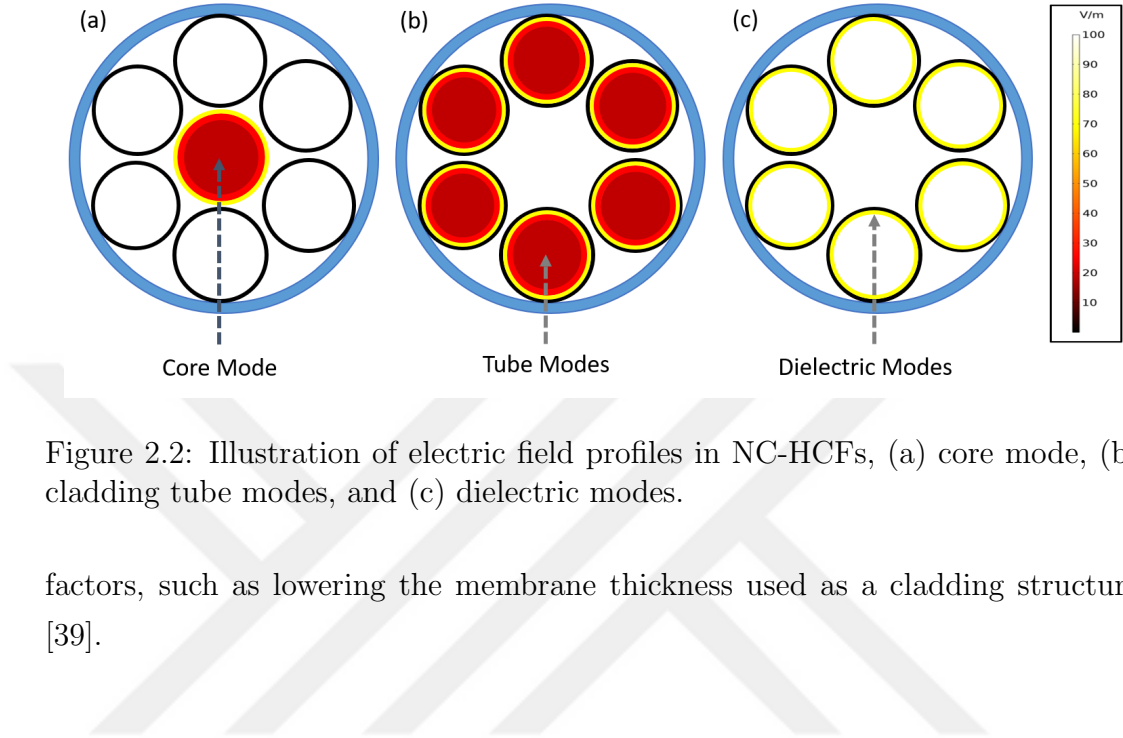


Figure 2.2: Illustration of electric field profiles in NC-HCFs, (a) core mode, (b) cladding tube modes, and (c) dielectric modes.

factors, such as lowering the membrane thickness used as a cladding structure [39].

2.2 Fabrication Method of HCMPOF

2.2.1 Preform Fabrication

To achieve fibers, much of the effort is put into the primary process of preform fabrication. A preform represents the macro size of the fiber geometry. Among different methods, drilling was applied first for fabricating MPOFs, where a non-hexagonal shape can be given, unlike the stack and draw process [14]. Yet, drilling length and surface roughness become the main limitation in this method [59, 86].

Using a second layer of polymer material instead of air holes in MPOFs comes with a solvent deposition technique [87]. Other methods, such as casting [88], billet-extrusion [15], stack and draw [5, 89], 3D printing [10, 11, 12, 16], have also been investigated in mPOFs fabrication. Among these methods, stack and draw techniques and 3D printing have shown promising results for making hollow core polymer material-based performances. Figure 2.3 shows some of the preform fabrication process methods.

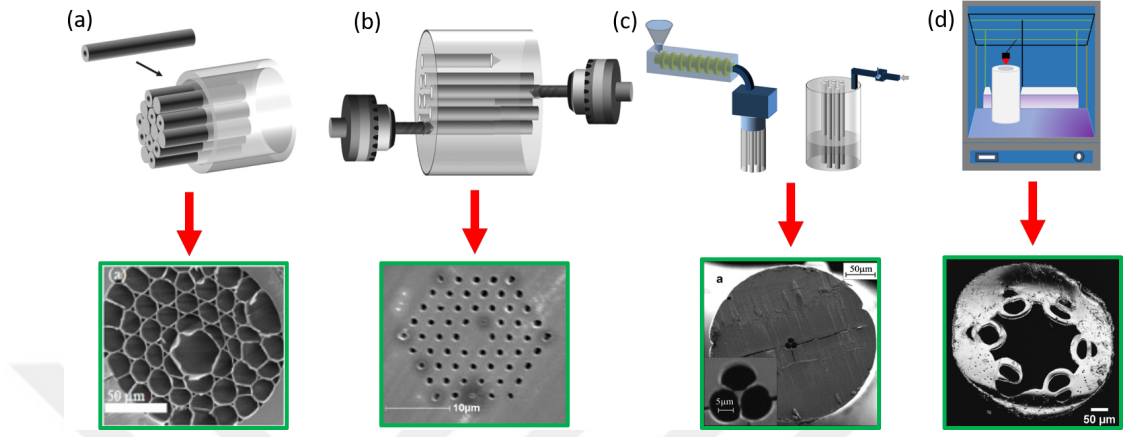


Figure 2.3: Different preform fabrication methods, (a) stack and draw [5], (b) drilling [14], (c) billet and extrusion [15], and (d) 3D printing process [16].

2.2.1.1 Stack and Draw Technique

The stack and draw process for polymer-based preform fabrication is taken from the silica optical fiber fabrication technique [2, 5, 47, 52, 89, 77, 85]. In this technique, hollow core capillaries of a desired thickness are drawn through the thermal drawing technique. These capillaries are stacked together inside another tube with supporting capillaries. Since these capillaries support the structure tightly, they are used in a shorter length on both ends of the preform. For example, suppose the preform length is 1 m. In that case, the supporting capillaries will be stacked along with a center capillary on both ends, only keeping the middle portion of the preform (50 cm) without any support [56]. After that, 3-5 mm outer diameter-based canes are drawn from this stacked preform of 15-20 mm outer diameter. Later, the fiber of 200-500 μm outer diameter is drawn from these canes. During the fiber drawing process, additional care is also taken. Figure 2.4 illustrates the preform fabrication method based on the stack and draw technique. However, this method possesses drawbacks regarding the geometry of the fiber. Fabrication of intricate geometry-based fiber is not feasible using this method [6, 10, 11, 13].

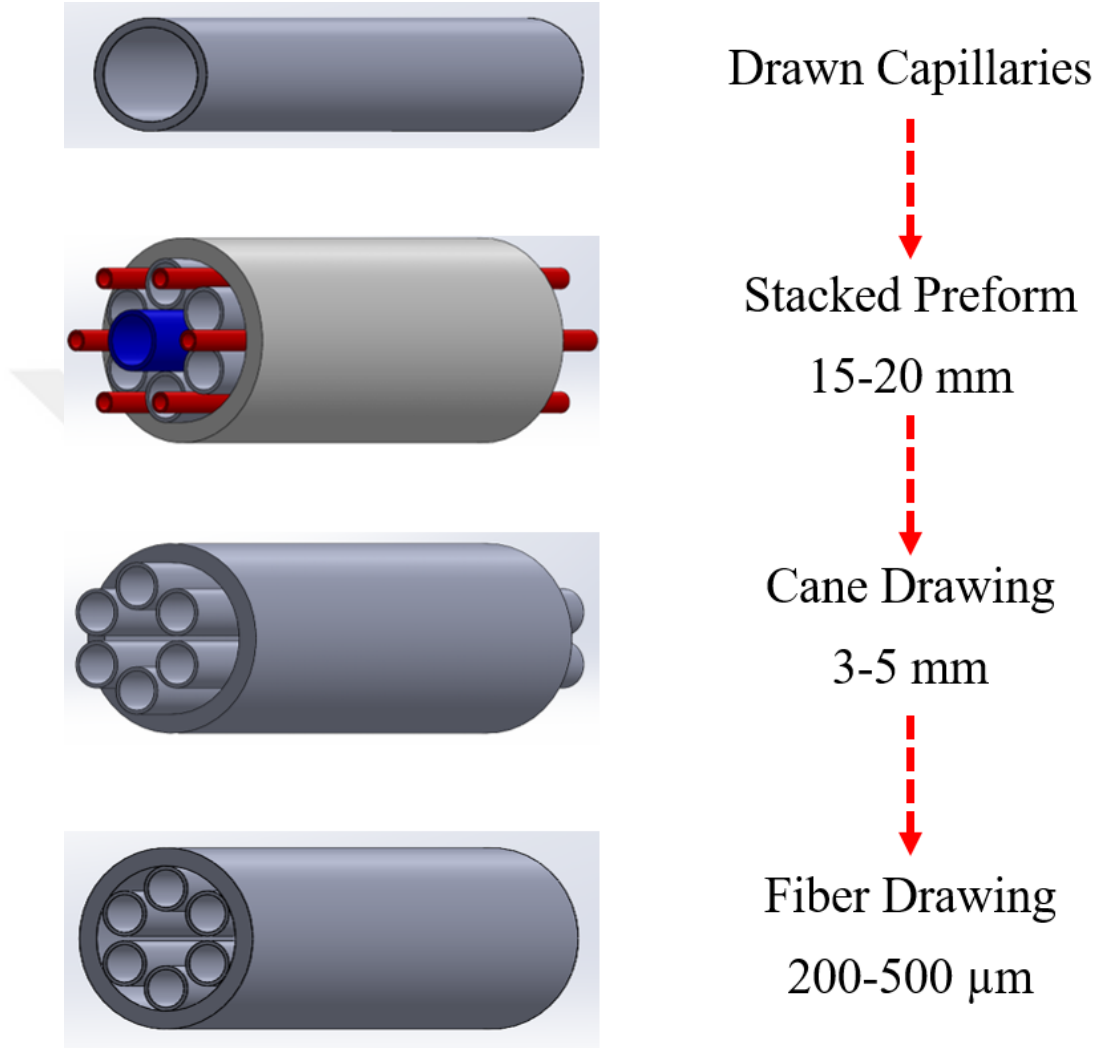


Figure 2.4: Schematic of different steps of preform and fiber fabrication process based on stack and draw technique.

2.2.1.2 3D Printed Preform Fabrication

Recently, additive layer manufacturing (ALM) or 3D printing has become more appealing to form complex designs due to its ease of use, lower cost, and less time requirements. The main advantage of 3D printing is the ability to fabricate complex structures while precisely controlling the features and dimensions. 3D printing has been involved in research by manufacturing microfluidic devices, energy storage, and sensors [90, 91, 92]. This approach has been recently explored to produce fiber-reinforced composites and optical fibers [16, 93]. Among different

types of additive layer manufacturing (ALM) techniques, such as stereolithography [94], powder bed fusion (PBF) [95], selective laser sintering (SLS) [96, 97], binder jetting (BJ) [98], etc., fused deposition modeling (FDM) has earned more popularity due to its availability, ease of use, variety of materials' option to choose from and for its lower cost and less time-consuming process [16, 99, 100].

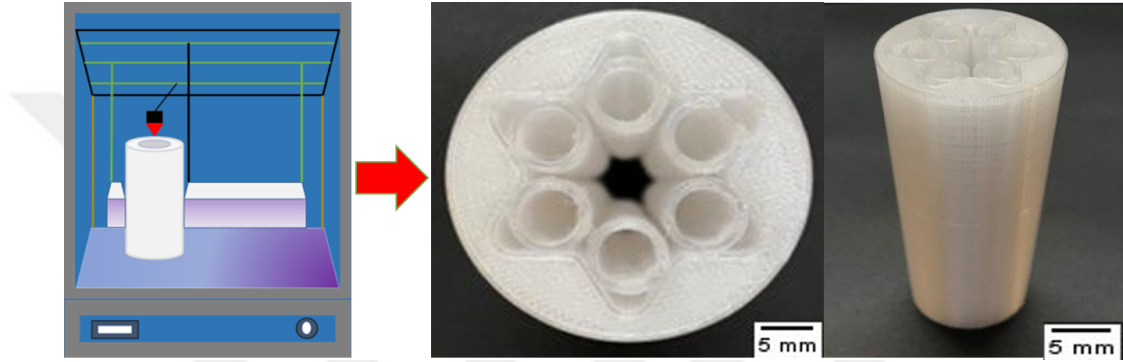


Figure 2.5: Schematic of optical fiber preform fabrication process via FDM technique [13].

In the FDM technique, structures are built by depositing materials layer by layer through the melt extrusion process. Using this technique, any intricate fiber geometry-based preform can be produced. During FDM based 3D printing process, mechanical and optical properties of extruding material, extrusion and bed build plate temperature, printing speed, layer height and thickness, and printing pattern play vital roles in achieving better preform structure [8, 9, 10, 11, 12, 13, 16, 99]. Since in HCFs structuring the outer cladding geometry in an engineered way can provide lower confinement loss, different intricate fiber geometry have already been proposed [13, 57, 58, 100, 101]. But the fabrication feasibility of these fibers hasn't been resolved yet. FDM can manufacture these complicated designs without going through a time-consuming stack and drawing process. Furthermore, the necessity of cleanroom and expensive manufacturing process can be avoided. Figure 2.5 shows a schematic of the 3D printing process for optical fiber preform fabrication.

2.2.2 Thermal Drawing of HCMPOF

After preparing the preform, achieving similar fiber geometry in the microstructured level is crucial. A thermal drawing tower is used to fabricate optical fibers. Figure 2.6 represents the entire process of fiber fabrication from a preform. At first, the prepared preform is fed into a radiative furnace, where it reaches a certain glass transition T_g temperature and becomes soft enough to elongate instead of melt. This fluid mechanical behavior is utilized in the fiber drawing process. Inside the furnace, the preform starts to form a neck region, and with the help of gravitational force, the fiber is collected in a spool. Several parameters are considered during this process, such as preform feed speed $v_{feed}^{preform}$, capstan speed $v_{capstan}^{fiber}$, drawing temperature $T^\circ\text{C}$, and drawing tension. Preform feed speed and capstan speed specifically determine the collected desired fiber diameter and can be given as follows:

$$d_{fiber} = d_{preform} \frac{\sqrt{v_{feed}^{preform}}}{\sqrt{v_{capstan}^{fiber}}} \quad (2.2)$$

Here, d_{fiber} represents the diameter of the drawn fiber from the diameter of the preform ($d_{preform}$), whereas $v_{feed}^{preform}$ gives the feeding speed of the preform inside the furnace and $v_{capstan}^{fiber}$ is the pulling speed of the fiber from the capstan.

Moreover, the achievable fiber length from a prepared preform can also be determined using another equation as follows:

$$l_{fiber} = l_{preform} \left(\frac{d_{preform}}{d_{fiber}} \right)^2 \quad (2.3)$$

Here, l_{fiber} represents the achievable fiber length from a prepared preform $l_{preform}$, where $d_{preform}$ and d_{fiber} are the preform diameter and the fiber diameter, respectively.

The importance of drawing temperature and drawing tension is also significant, along with the fiber diameter and total fiber length. Especially while drawing a

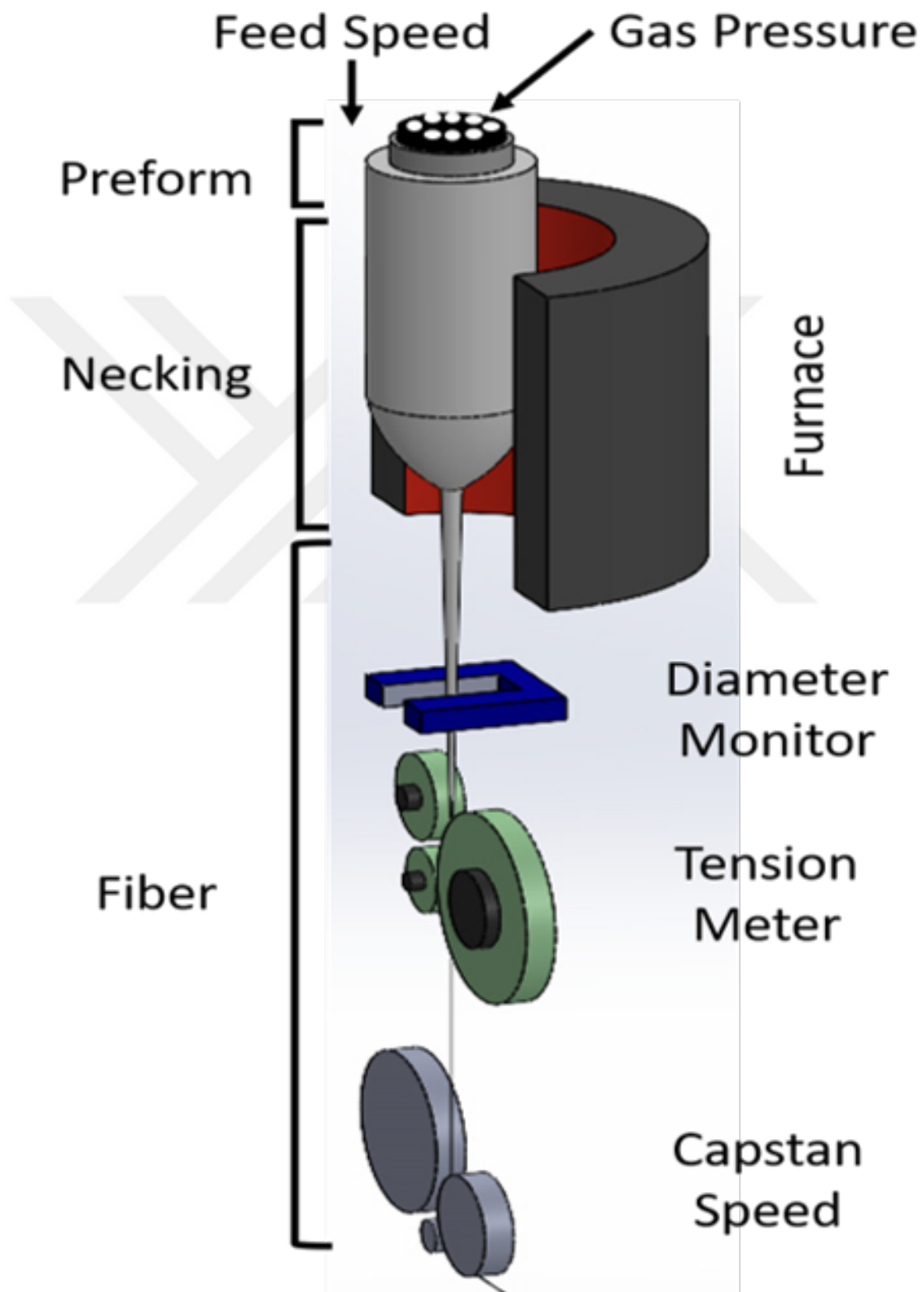


Figure 2.6: Schematic of thermal drawing process from preform to fiber.

micro-structured fiber, these parameters play a vital role. If the fiber is drawn at high viscosity at a reduced temperature, then there remains a possibility of preform breakage. Again, fiber drawn at low viscosity and high drawing temperature from a preform comes with a collapsed geometry in micro-structured optical fiber drawing, which is also undesirable. Proper control and consideration of these parameters during the fiber drawing process are crucial to achieving desired fiber geometry with a uniform diameter. Furthermore, considering the surface tension acting on the capillaries of micro-structured fiber drawing, external gas pressure can also be applied in the core and cladding region of the preform [5, 89, 100]. A tension meter and fiber diameter monitor are also necessary tools to monitor the whole drawing process, as shown in Figure 2.6.

2.3 Optical Loss Measurement

After successfully fabricating the optical fiber, it requires an optical measurement to find the propagation loss, bending loss, modal property, numerical aperture (NA), chromatic dispersion, cutoff wavelength, number of existing modes in the fiber, etc.

2.3.1 Transmission Loss Measurement Optical Setup

The optical setup for straight transmission loss measurement of a fiber is shown below:

A supercontinuum (SC) laser with an emission spectrum ranging from 450 to 2400 nm (YSL Photonics SC-5, China) is collimated using a microscopic lens. Then collimated light is coupled into a fiber using another microscopic lens. During the light collimation as well as coupling into the fiber, NA matching plays an important role in maximizing the light guidance efficiency and minimizing the loss due to coupling inefficiency. Then collected light from the other fiber end is

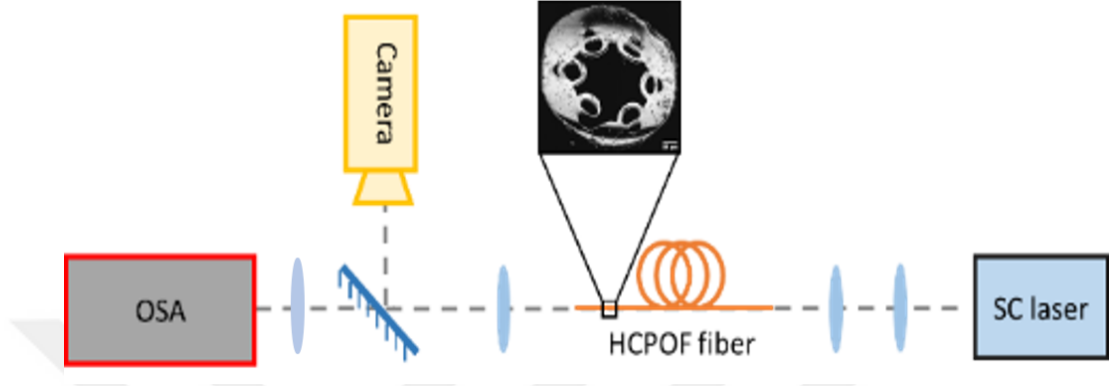


Figure 2.7: Optical setup for straight transmission loss measurement in a HCM-POF [13].

again collimated and coupled into the Optical Spectrum analyzer (OSA) (Yokogawa AQ6370B, Japan) using another two sets of microscopic lenses. The output power of the fiber end at each length is collected from OSA. These lenses are set up over 3D stages to maximize their freedom of movement to achieve high-efficiency coupling. Meanwhile, a laser beam profiling camera is also added to the setup to monitor the output images of the fiber at different fiber lengths with the help of a movable reflecting mirror. The cutback measurement method is used for straight transmission loss measurement to minimize the effect of the coupling problem. In this method, a fiber is cut at different lengths after collecting their optical spectra at each length. Then, straight transmission loss is measured using the equation given below:

$$\alpha_{loss} = \left(\frac{10}{L} \right) \left(\frac{P_{in}}{P_{out}} \right) \quad (2.4)$$

Here, α_{loss} represents the transmission in dB/unit length. P_{out} and P_{in} gives the measurement of the output and input power collected from the fiber length at L_1 and L_2 respectively, where $L = L_1 - L_2$ in unit length. The transmission loss of Silica optical fiber is given in dB/km unit. Besides the propagation loss along the fiber, other factors, such as splicing loss, micro and macro bending loss, and coupling loss, also play a crucial role. To minimize the coupling loss in our optical loss measurement of mPOFs, we couple the light using lenses of having similar NA. The fiber was kept straight as much as possible to reduce the micro

and macro bending-induced losses. Fiber was cut at several lengths to minimize the splicing-initiated loss.

2.3.2 Bend Loss Measurement Optical Setup

Optical fibers face different mechanical effects; hence, light propagation loss increases due to the fiber's bending (micro and macro). Sometimes installing the fiber in the field at some instances, we need to bend the fiber. While bending the fiber at different radius it also increases the transmission loss. So, bend loss measurement is also required to determine the optical fiber's characteristics. Figure 2.8 shows the optical setup for bend loss measurement of our MPOFs below:

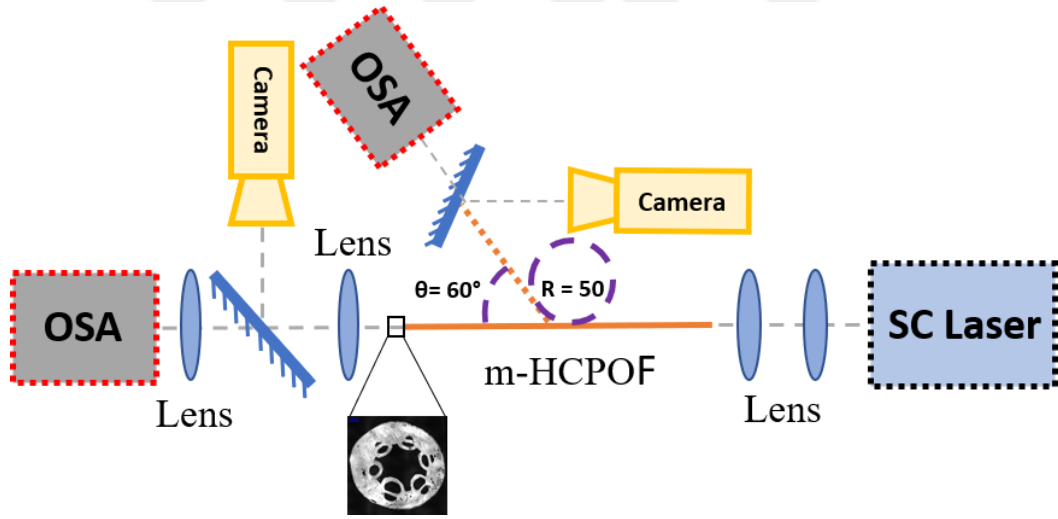


Figure 2.8: Optical setup for bend-induced loss measurement under mechanical effects in a HCMPOF.

Initially, the optical power with the transmission signal is collected from OSA for a straight long fiber using the similar optical setup as mentioned in figure 2.3.1.1. Since POFs possess huge losses compared to silica optical fiber (especially in our fabricated mPOFs), we tried to extract the bending loss data of those fibers at different bending angles (30° , 45° , 60° , 90° , 120° , 135°) in different bending radius (25, 50 mm). After the initial straight transmission measurement, the fiber was bended at those different angles for a certain bending radius and with

the modified setup the transmission spectra were collected. After the collection of data for different bending the fiber was cut into shorter length the measure was further taken by the similar optical setup shown in the figure 2.8. Bend loss measurement can be obtained in an analogous way using the equation 2.4.

2.4 Fiber Characterization

2.4.1 Axio Scope A1 Laboratory Microscopy

For continuous use during fiber drawing and optical characterization, the axio scope A1 laboratory microscope is a great tool in a laboratory environment. Especially someone who needs to observe the structure and run the microscope for a long period. Since MPOF geometry deforms easily, several cuts are needed during the cut-back measurement method. For this purpose, imaging through this microscope becomes user-friendly to observe the proper cut (without or with minor deformation) during optical characterization. Several contrast methods, such as dark and bright field, phase contrast, and fluorescence, according to the user's demand.

2.4.2 Laser Scanning Confocal Microscopy

For the imaging of HCmPOFs profiling, a laser scanning confocal microscope (LSCM) (Keyence VKX 100, Japan) is used. The laser beam is converged and coupled to a sample's surface in this microscopic method. The reflected light is collected and further refocused in a pinhole. The pinhole is situated in front of the detector, which detects the intensity of the incoming light through the pinhole. To achieve the optimum intensity during imaging, the density of the laser illumination should match the acceptance of the pinhole detector. The measured intensity is high when the laser's confocal volume intersects the surface. But sometimes, we observe lower intensity-based imaging because the confocal

volume does not cross the surface. While scanning the sample's surface area, the relative position between the objective lens and the surface is important.



Chapter 3

Stack and Draw technique based HCMPOFs

3.1 Hollow Core Preform Fabrication

Preform fabrication is a significant step for fabricating stack and drawing technique-based HCMPOF. To fulfill the criteria, commercially available 30 cm long polycarbonate (PC) sheets were first rolled over Teflon tubes. Figure 3.1 (a) depicts the polymer rolling process. The reason for using Teflon tubes is that even at higher temperatures (200° - 350°C), the polymer doesn't stick with the Teflon. A 14.8 mm outer diameter (OD) Teflon tube that acted as an inner diameter (ID) of the hollow PC preform was chosen for our work. The polymer was rolled over the Teflon until 18 mm OD was reached with a thickness of 1.6 mm. The reason for choosing this particular diameter is to maintain the draw-down ratio of around 10.9 from preform to fiber. Because according to our HCPOF preform design, the $\text{OD} = 1.65$ mm and $\text{ID} = 1.35$ mm with a thickness of 0.15 mm capillaries were required for the stacking process. Extra care was taken during the polymer rolling process to prevent the formation of trapped air gaps inside the rolled polymer layers. Otherwise, air bubbles can be observed after the consolidation process, leading to nonuniform fiber diameter during the fiber drawing process and hence

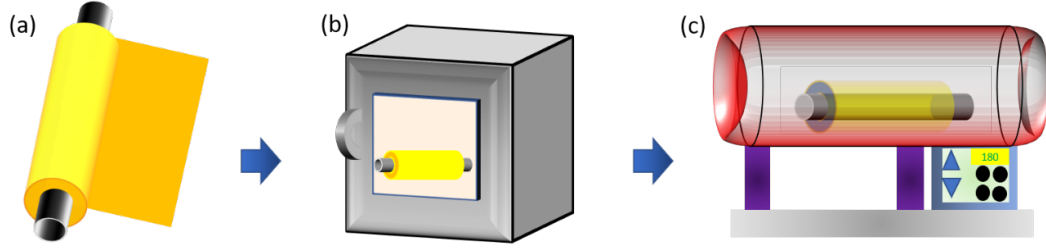


Figure 3.1: Schematics of the preform fabrication process (a) polymer rolling, (b) vacuuming at elevated temperature, and (c) consolidation of the rolled polymer preform.

perform breakage. Moreover, Teflon tape was wrapped over the rolled polymer to hold the structure without deformation during consolidation. Since most of the polymer materials have an affinity to air moisture, the rolled preform was kept inside a vacuum chamber, as shown in Figure 3.1 (b). The preform was heated inside the chamber at an elevated temperature (90°C). The elevated temperature was chosen way below the polymer material's glass transition temperature (T_g) so the structure doesn't deform.

Later, the preform is consolidated inside a custom-made horizontal and dual-zone radiative furnace, as shown in Figure 3.1 (c). The rolled polymer possesses some air gaps between the rolled layers and does not stick together, which is unsuitable for successful fiber drawing. In this process, the temperature is raised way beyond the material's glass transition temperature, where polymer materials soften enough and stick to each other, forming a solid preform. For our work, at the first stage, the temperature was raised at a rate of $10^{\circ}\text{C}/\text{min}$ from ambient condition to 140°C inside the furnace and kept at that temperature for 2 hours 20 minutes, which is still lower than the glass transition temperature of PC ($T_g = 147^{\circ}\text{C}$). In the second stage, the temperature was raised to 185°C and kept for 45 minutes. At this point, the polymer layers become soft enough to stick together and form a solid, glassy type transparent structure. The process is carried out using a predefined manual and under vacuum conditions. After that, the preform is cooled to the ambient temperature and kept inside the vacuum chamber at an elevated temperature again.

3.2 Thermal Drawing of Capillaries

A custom-made fiber draw tower with a dual zone radiative furnace was used to draw capillaries of desired diameters from the fabricated preform. During the capillary drawing process, consideration of parameters, such as preform feed speed, capstan speed, and achievable fiber length, can be precalculated using equations 2.3 and 2.4, where the length and diameters of the preform and desired diameters of the capillaries are already known. The fiber was drawn at 230^o C temperature with preform feed speed and capstan speed of 3.5 mm/min and 0.32 m/min, respectively, as depicted in Figure 2.6. But no gas pressure was applied during the capillary drawing process. Figure 3.2 shows the image of the drawn capillary taken by Axio Scope Laboratory A1 Microscope.

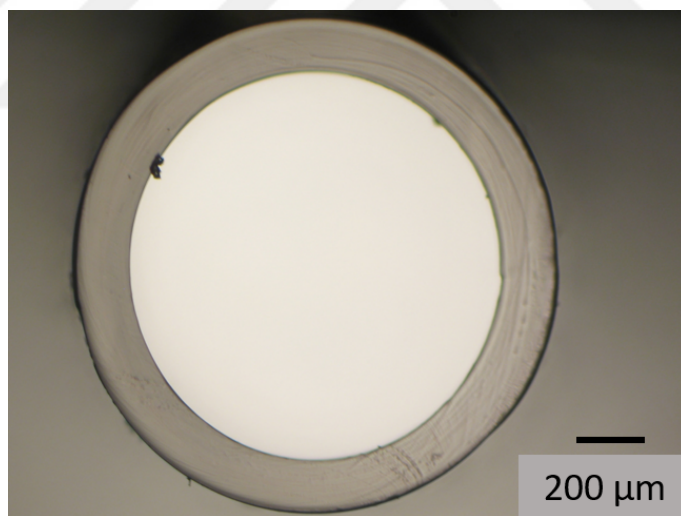


Figure 3.2: Thermally drawn hollow core capillary from the hollow core preform of PC material.

3.3 Preform Stacking

For five tubes and six tubes-based tubular HCMPOFs, preforms were stacked with five and six main capillaries drawn from the fiber tower, respectively. Generally, the core-to-cladding diameter ratios for HCMPOF preforms design are kept at

2:1 and 1.5:1. The capillaries were stacked inside a 30 cm long consolidated outer jacket made of PC materials following the process mentioned in Figure 3.1. The ID and OD of the outer jacket are 6.63 mm and 14.2 mm (Figure 3.3 (a)). As shown in Figure 3.3 (b), those drawn capillaries were stacked inside with a center support of 3.3 mm OD and 5 and 6 other supporting capillaries to hold the structure for the five-tube and six-tube base HCMPOF preform, respectively. The center capillary with other supporting capillaries is stacked just at the preform-ends. In the mid-section, main capillaries only exist, as Figure 3.3 (c) shows. After the stacking process, the holes of the supporting capillaries and center holes are blocked with resin hardener to prevent gas flow through those regions.

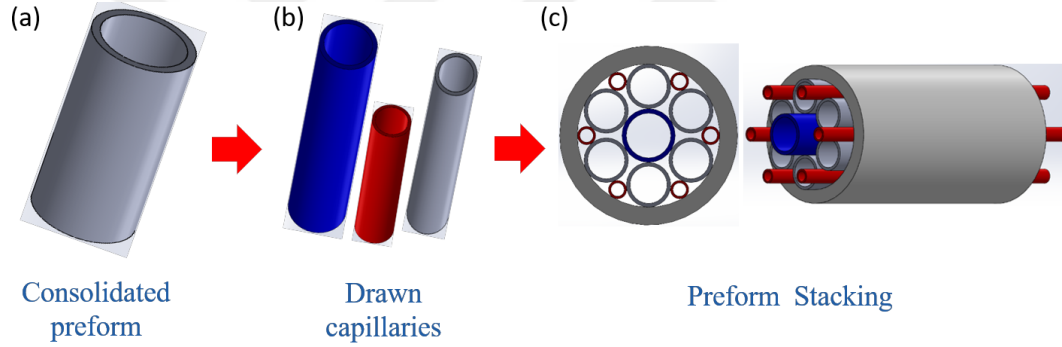


Figure 3.3: Schematics of preform stacking process, (a) Prepared outer jacket for stacking, (b) drawn capillaries, (c) Stacking main capillaries inside an outer jacket with supporting capillaries.

3.4 Fiber Drawing of Stacked Preform

The prepared stacked preform was inserted inside the same custom-made fiber draw tower with a dual zone radiative furnace in this step. During the HCMPOF drawing process, consideration of parameters, such as preform feed speed, capstan speed, and achievable fiber length, can be precalculated using equations 2.3 and 2.4, where the length and diameters of the preform and desired diameters of the fiber are already known. The fiber was drawn at 234^o - 236^o C temperature, as shown in Figure 2.6. After a certain point, the preform starts to form neck

inside the furnace and comes down due to the gravitational force. The fiber was connected to the capstan at a 1.25 m/min drawing speed. The fiber was directly drawn from the preform with a preform feed speed of 2.5 mm/min at a high draw tension (70-80 g). During the fiber drawing process, the temperature decreased to 4° C to make the material more viscous. This process also helps to prevent the effect of preform micro-structured holes' collapse. Additionally, 50 ccm air pressure is applied specifically through the hollow capillary region to inflate them and to hold the micro-structures inside the fiber.

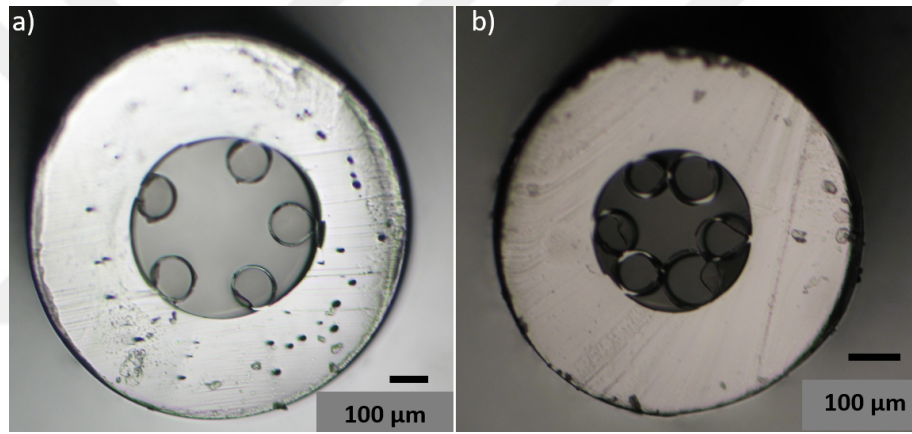


Figure 3.4: Cross-sectional images of the (a) five-tubes and (b) six-tubes based HCMPOFs.

The final outer diameters of the five-tubes and six-tubes tubular HCMPOFs were around 475 μm . Figure 3.4 (a) and (b) depict the cross-sectional images of the five tubes and six-tubes tubular HCMPOFs. The five-tubes based HCMPOF consists of a $153.1 \pm 8 \mu\text{m}$ core diameter and $74.5 \pm 3 \mu\text{m}$ capillary diameter surrounding the core having thickness of $4.5 \pm 0.3 \mu\text{m}$ on average. The geometry is not well confined, and the distance between two adjacent capillaries is very high, the main contributor to the light leakage in HCFs. On the other hand, six-tubes tubular HCMPOF consists of a $68.53 \pm 5 \mu\text{m}$ core diameter and $52.8 \pm 4 \mu\text{m}$ capillary diameter surrounding the core having thickness of $6.34 \pm 0.5 \mu\text{m}$ on average. In this case, the core is relatively well structured compared to the five tubes based simple tubular fiber.

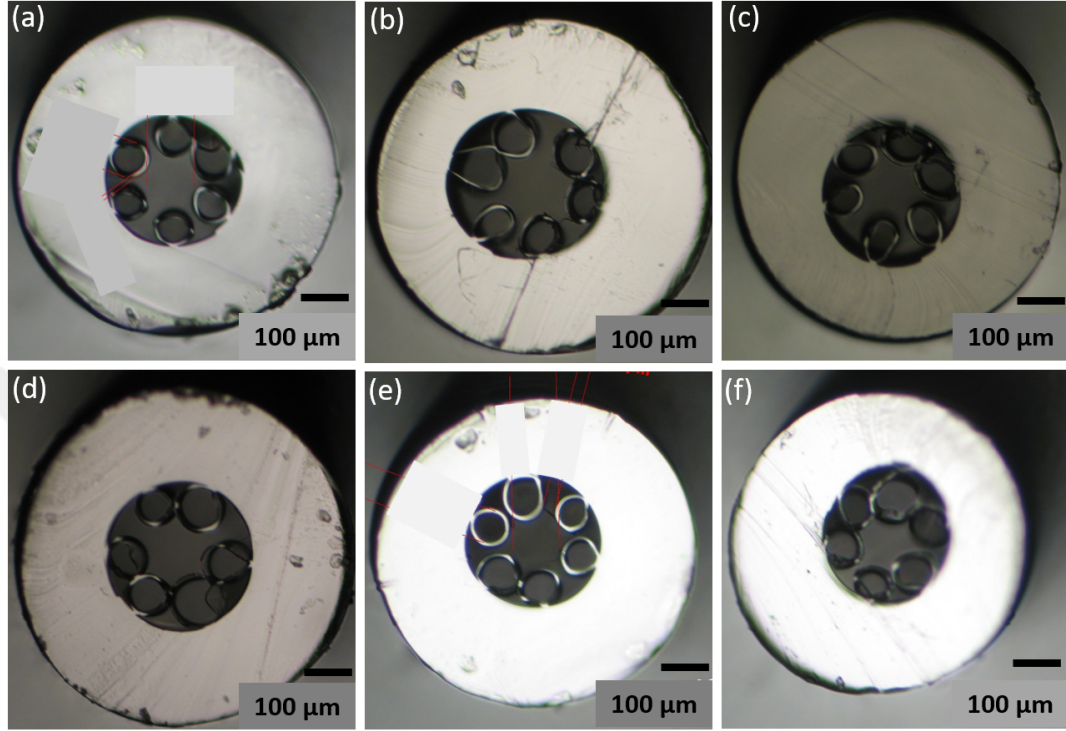


Figure 3.5: Comparison of the cross-sectional images of the drawn fiber at different fiber lengths (a) 104 cm, (b) 91 cm, (c) 76 cm, (d) 56 cm, (e) 38 cm, and (f) 27 cm from the same batch of 104 cm length.

A detailed investigation was carried out on 104 cm long six-tubes based HCM-POF, as shown in Figure 3.5 (a)-(f). The fiber was cleaved at different fiber lengths to observe the condition of the capillaries inside the MPOF. As mentioned, the figure shows that the capillaries' positions inside the fiber are shifted throughout 104 cm long fiber at different lengths. Even the change in the position of the fiber capillaries' can be observed at a 15 cm distance. This has a negative impact on the propagation spectra for the loss measurement of the drawn fiber.

Since polymers are not rigid compared to silica material, especially at lower thickness, they possess significant bending issues, as shown in Figure 3.6. Even in shorter lengths, polymer bends a lot compared to silica. The setup of the capstan in the fiber draw tower also plays a significant role in bending these capillaries. Thus, stacking those capillaries inside an outer jacket creates a shift in the position of the capillaries inside the preform. The supporting and center capillaries are only at both sides of the preform end. So, these are insufficient to

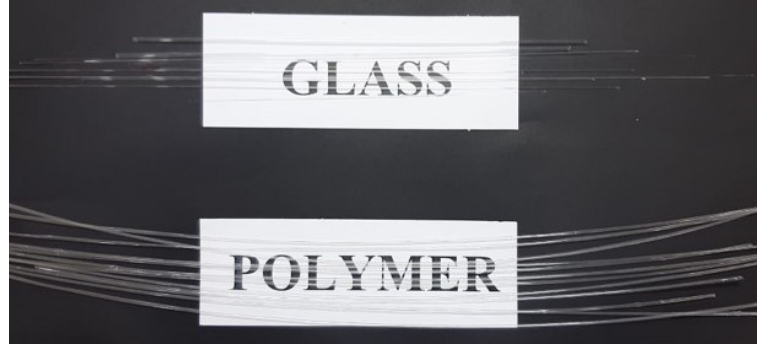


Figure 3.6: Bending issues with polymer capillaries compared to silica capillaries (25 cm).

keep those capillaries in a fixed position.

3.5 Propagation Loss Measurement

3.5.1 Loss Measurement of Five-Tubes based Tubular HCMPOF

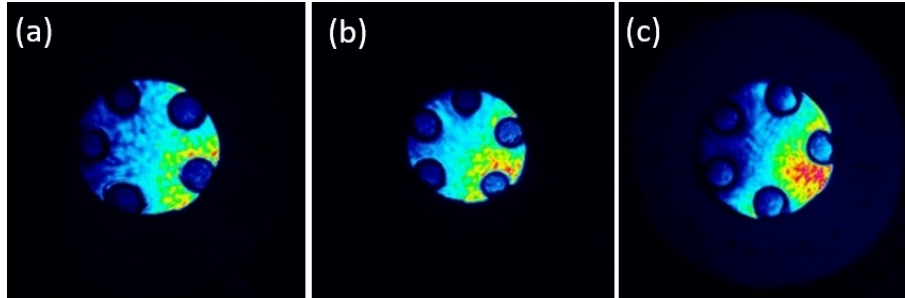


Figure 3.7: Output images of the five-tubes tubular HCMPOF, when light is coupled to the core at (a) 27 cm, (b) 20 cm, (c) 15.1 cm lengths, respectively.

The straight transmission loss measurement for five-tubes tubular HCMPOF was carried out using the optical setup depicted in Figure 2.7. A 27 cm long fiber was used for the cutback measurement method. The light guidance through the core can be observed in Figure 3.7 at different fiber lengths. The large size of the core with speckles observed from the images significantly proves the multimode

nature of the fiber.

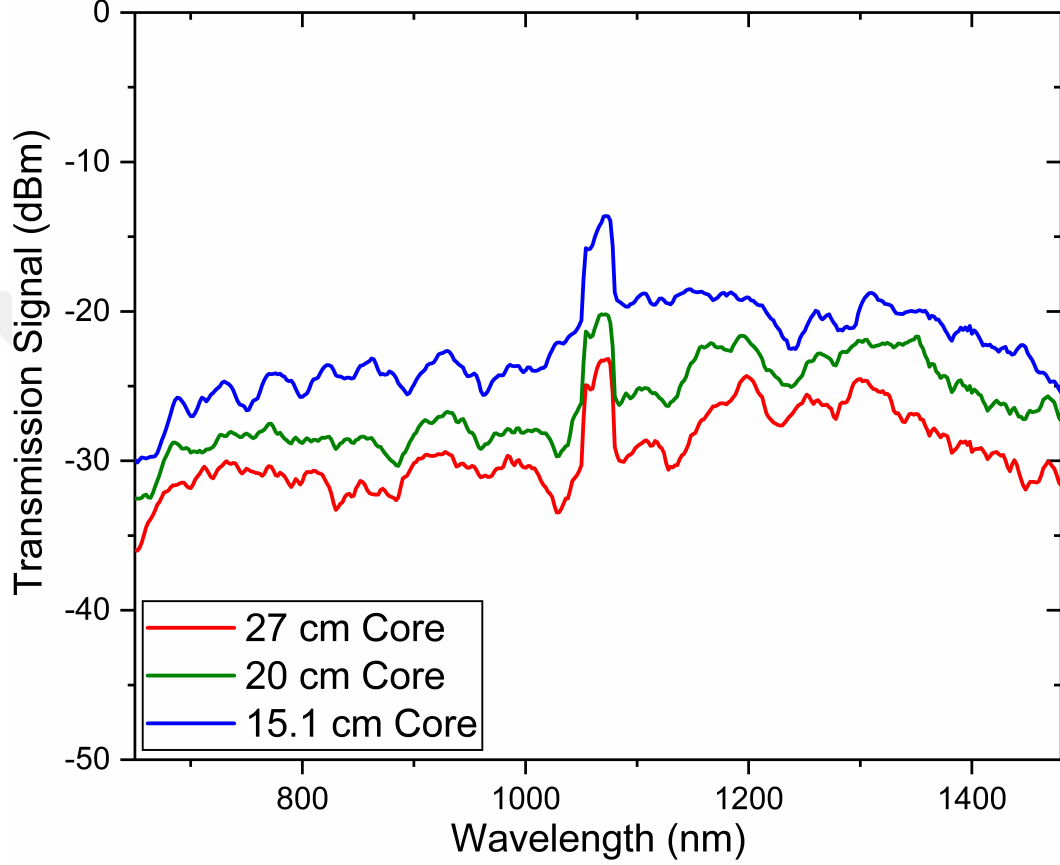


Figure 3.8: Transmission signals of the core guidance of the drawn fiber at different lengths.

To confirm the light guidance through the hollow core, transmission spectra at several fiber lengths (27, 20, and 15.1 cm) were collected by an OSA. Figure 3.8 shows the transmission signal spectra of the core guidance with the aforementioned lengths from 650 to 1500 nm wavelength ranges. It can be observed that the transmission spectra for the 27 cm and 20 cm are similar. But a slight change is visible for the 15.1 cm long fiber length transmission spectrum. Since the gap between the capillaries of the 15.1 cm long fiber was larger than the other fiber lengths, the variation in the transmission signal is significant. Again, these gaps between those capillaries are the major reason for higher leakage loss despite its bigger core size, as shown in Figure 3.9.

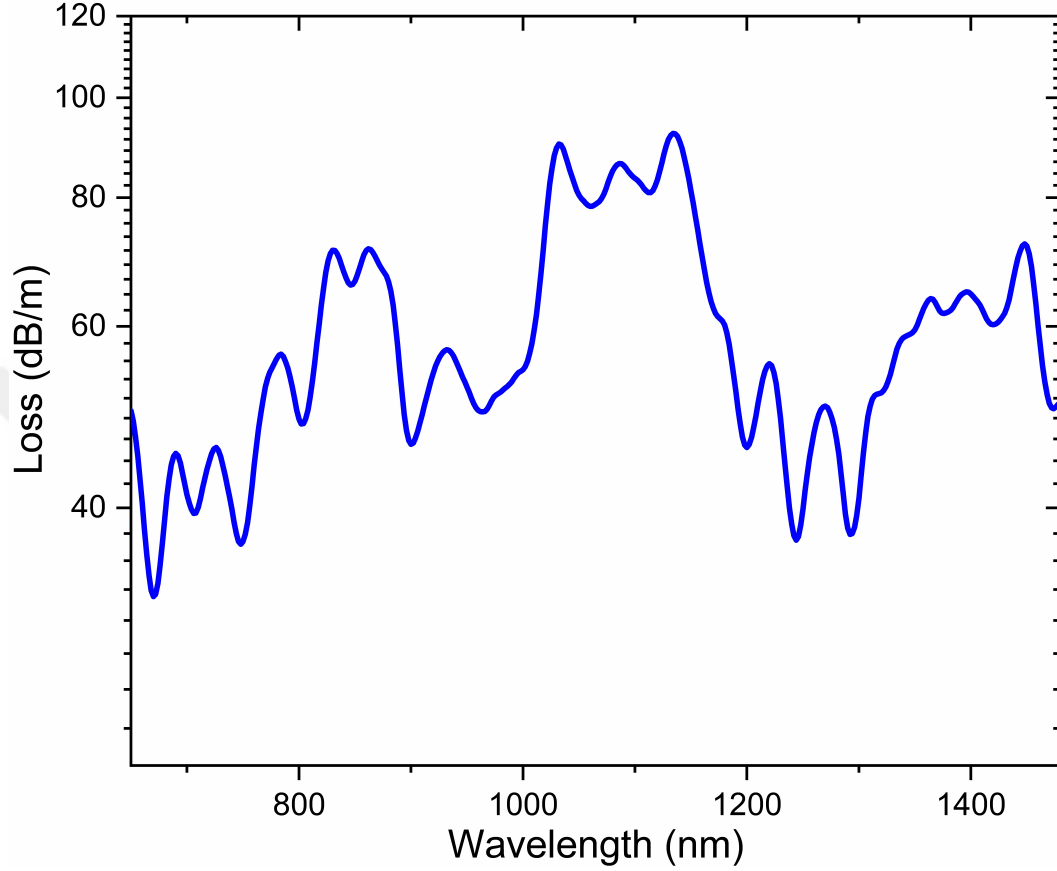


Figure 3.9: Measured optical propagation losses of five-tubes tubular HCMPOF between 650 nm to 1500 nm.

The cut-back method was used for the propagation loss measurements. Since the laser source is pumped at $1.064 \mu\text{m}$ wavelength, the high loss comes from there despite high transmission signals visible at that regime. The lowest propagation loss of 32.81 dB/m was achieved at 670 nm with an average optical loss of 51.78 dB/m between $6.5 \mu\text{m}$ to $1 \mu\text{m}$. Furthermore, the loss between $1.1 \mu\text{m}$ to $1.50 \mu\text{m}$ was averagely found as 58.95 dB/m (Figure 3.9). Despite having a thinner capillary thickness ($4.5 \pm 0.3 \mu\text{m}$) due to anomalous transmission signal, no significant resonance band was observed. The multimode guidance of the fiber with higher light leakage probability due to the failure to keep the geometry confined is the major reason for the higher loss in the NIR region despite the larger core geometry.

3.5.2 Loss Measurement of Six-Tubes based Tubular HCMPOF

The straight transmission loss measurement for six-tubes tubular HCMPOF was carried out using the similar optical setup depicted in Figure 2.7. A 44.7 cm long fiber was used for the cutback measurement method. The light guidance through the core can be observed in Figure 3.10 at different fiber lengths. The speckles inside the core observed from the images significantly prove the multimode nature of the fiber.

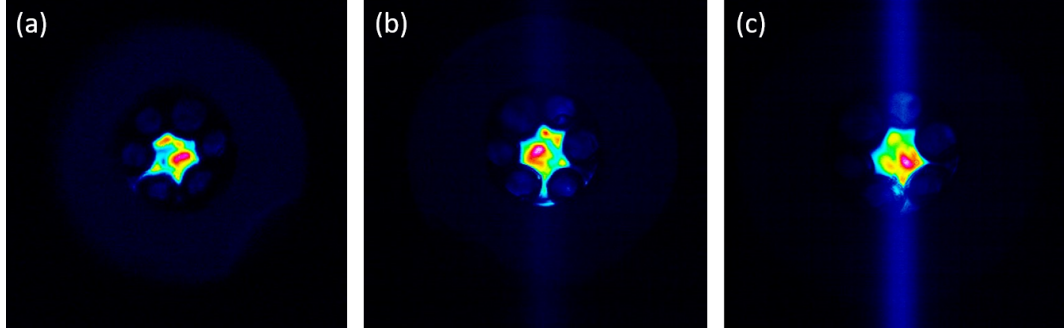


Figure 3.10: Output images of the six-tubes tubular HCMPOF, when light is coupled to the core at (a) 44.7 cm, (b) 33.7 cm, and (c) 17.5 cm lengths, respectively.

To confirm the light guidance through the hollow core, transmission spectra at several fiber lengths (44.7, 33.7, and 17.5 cm) were collected by an OSA. The light was also intentionally coupled to the 44.7 cm fiber's cladding to determine the guidance characteristic of the drawn polymer. Figure 3.11 shows the transmission signal spectra of the core and cladding guidance of the fiber with the aforementioned lengths from 650 to 1650 nm wavelength ranges. Although the transmission spectra for the fiber's core and cladding follow a similar trend between 0.65 and 0.8 μm , where the bulk polymer is transparent, a noticeable shift occurs at longer wavelengths due to the material absorption. The transmission behavior of the fibers in these areas supports that light is guided through the hollow core between 1.1 and 1.65 μm .

The cut-back method was used for the propagation loss measurements. Since

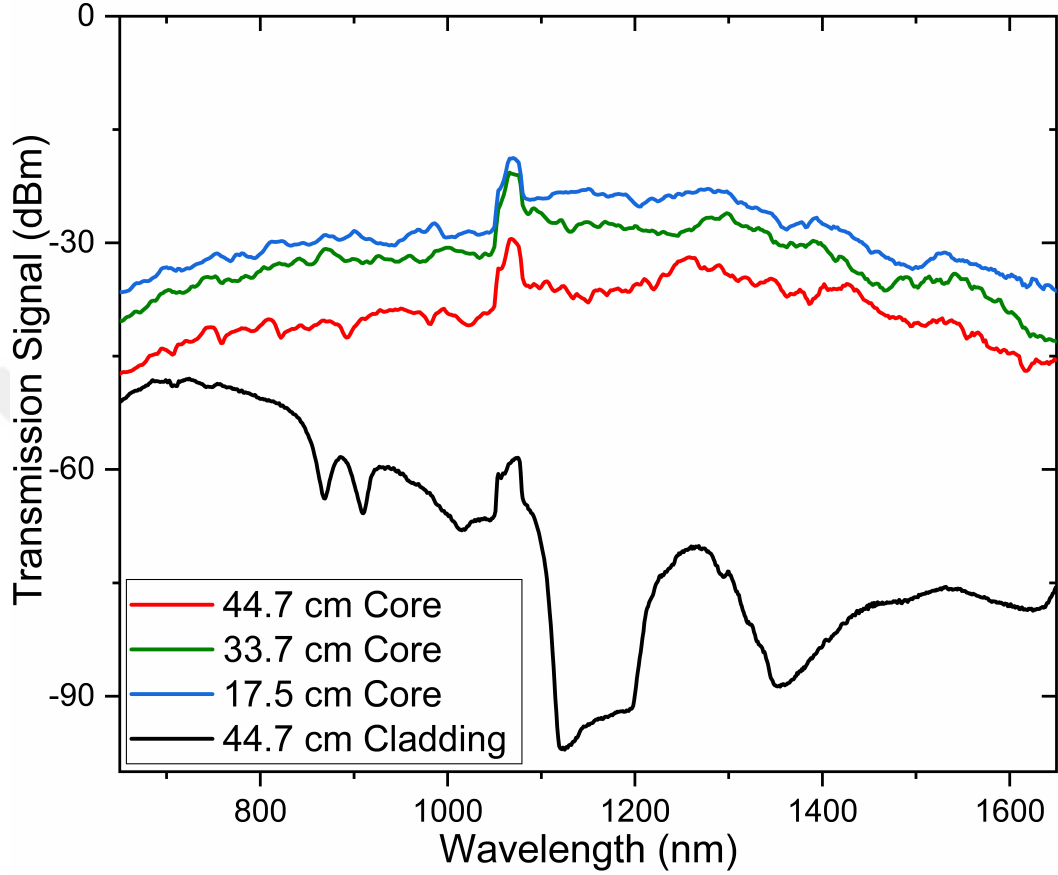


Figure 3.11: Transmission spectra for core guidance of the drawn fiber at three different lengths (44.7, 33.7, and 17.5 cm).

the laser source is pumped at $1.064\ \mu\text{m}$ wavelength, the high loss comes from there despite high transmission signals visible at that regime. The lowest propagation loss of $25.18\ \text{dB/m}$ was achieved at $1430\ \text{nm}$ with an average optical loss of $48.57\ \text{dB/m}$ between $6.5\ \mu\text{m}$ to $1\ \mu\text{m}$. Furthermore, the loss between $1.1\ \mu\text{m}$ to $1.65\ \mu\text{m}$ was averagely found as $49.40\ \text{dB/m}$ (Figure 3.12). Despite having a thinner capillary thickness ($6.34 \pm 0.5\ \mu\text{m}$) due to anomalous transmission signal, no resonance band was observed. The multimode guidance of the fiber with non-uniform light confinement and shifts of the cladding geometry, even in 15 cm lengths, are the major reasons for the higher loss in this wavelength region despite the smaller core diameter.

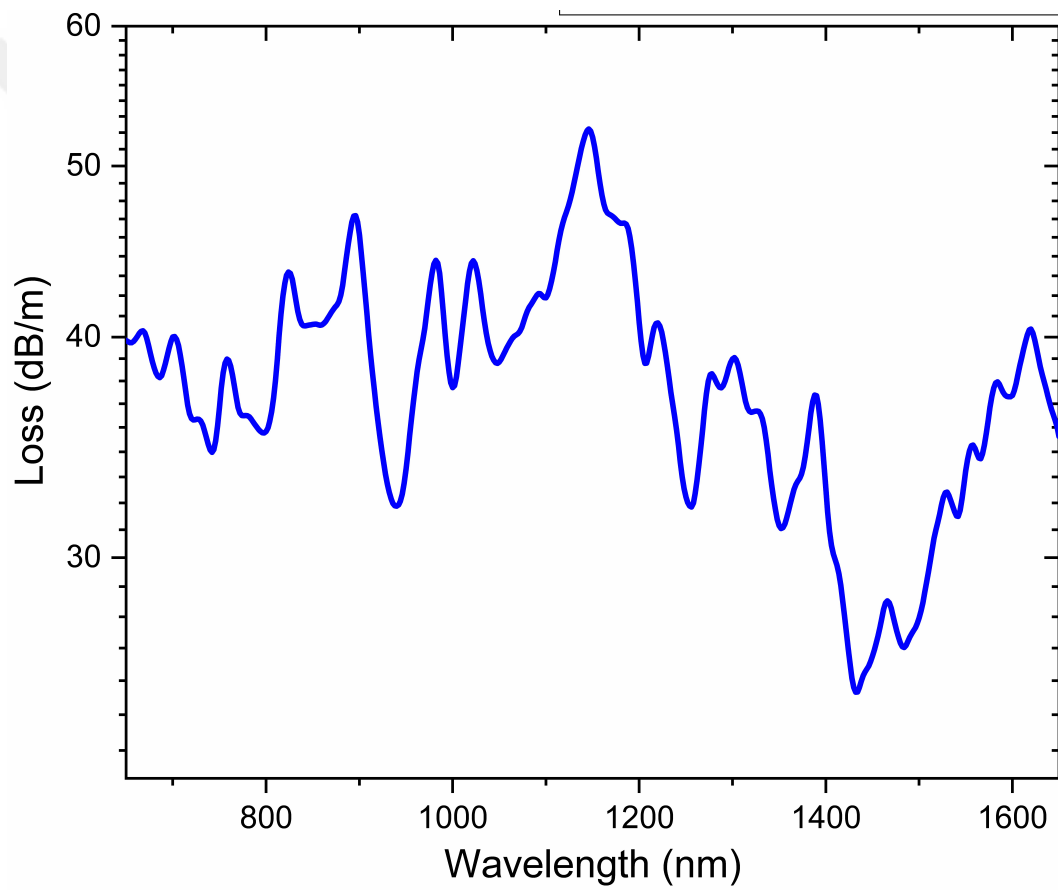


Figure 3.12: Measured propagation loss of six-tubes tubular HCMPOF between 650 nm to 1650 nm.

Chapter 4

3D Printed HCMPOFs

4.1 Preform Fabrication via Fused Deposition Modeling

The printing process and preform of the HCPOF with six-pointed star cladding tubes are shown in Figure 4.1 (a), (b). The design of the preform, and thus the HCPOF, was based on shaping the fiber's outer cladding to lower the transmission losses further [57]. A 175 mm long preform was printed as it was the maximum workspace of the FDM thermal 3D printer (Zortrax M200, Poland). The diameter of the preform was chosen to be $\phi = 38mm$, and it comprises evenly spaced six cladding tubes along the periphery (tube diameter $\phi_{ti} = 8.75mm$ and thickness $t = 0.80mm$) while surrounding the hollow core of the preform. The tubes are located at the pointed regions of the cladding, leaving additional air holes between the tubes and the outer polymer cladding to improve the light guidance. The extruder of the 3D printer was fed with a low-cost and commercially available translucent Z-Glass filament composed of PETG 80% fiberglass filler 12% and other additives 8% [13]. This material was given preference for its mechanical and chemical durability, UV resistance, and surface scratching. Furthermore, the 3D model can be printed precisely with this filament because of its lower shrinkage

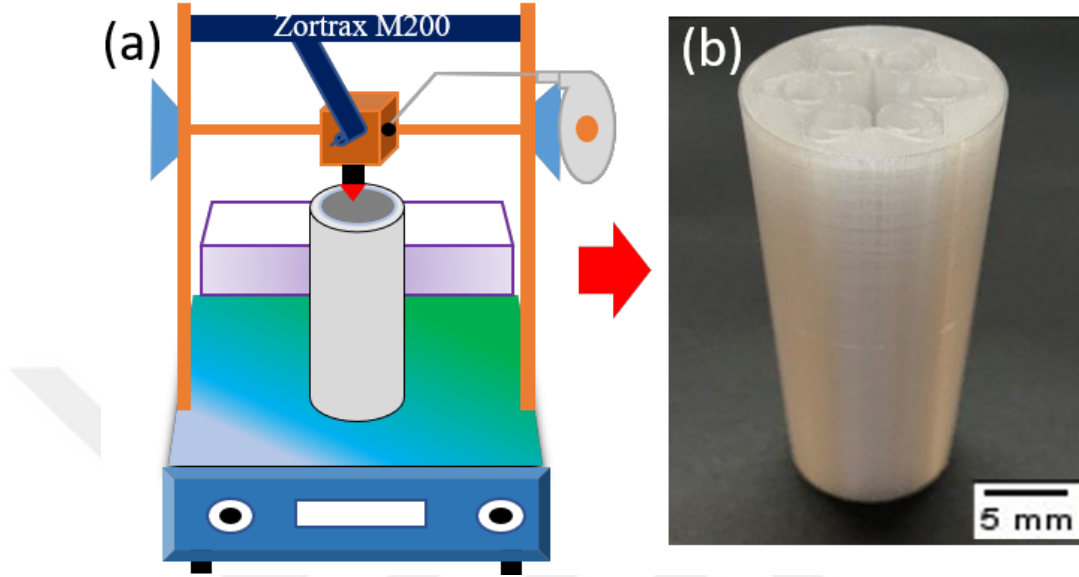


Figure 4.1: Preform fabrication process by ALM method (a) 3D printing process, and (b) perspective view of the hollow core preform.

rate than the other polymeric filaments used in 3D printers. The extruder and bed temperatures were set as $T_{ex} = 254^{\circ}C$ and $T_{bed} = 80^{\circ}C$ after the optimization studies on the printer. The thinnest possible layer height and thickness were selected as 0.14 mm and 0.8 mm, respectively. Since the printer nozzle deposits the polymer layer by layer at high extrusion temperature, it creates a chance of warping the solid structure. To avoid this problem, a solid infill pattern with 90% infill density was chosen, extending the printing time to $t = 32$ hours. Despite the translucency of the filament, insufficient polymerization with trapped air bubbles inside the printed structure caused the preform to be milky white after 3D printing. However, drawing into fiber cleared a considerable percentage of this opaqueness. The preform was prepared without going through the laborious and time-consuming intermediate steps of cane drawing and stacking.

4.2 Thermal Drawing from 3D Printed Preform to HCMPOF Fiber

The preform was prepared without going through the laborious and time-consuming intermediate steps of cane drawing and stacking [89, 5]. A custom-made fiber draw tower with a dual zone radiative furnace was used to draw fibers at 158°C with a capstan speed of 1.875 m/min, as schematically shown in figure 2.6. Fibers were directly drawn from the preform skipping the intermediate cane drawing step. The quick fabrication process performed in this work was achieved by selecting a mechanically robust filament and adjusting the drawing parameters. This selection creates an additional advantage of preserving the printed preform cross-section in the fiber since the cross-section was easily damaged with additional heating cycles [12]. Moreover, during the fiber drawing process, the propensity of the core and capillaries to expand and contract was observed respectively under low drawing tension since surface tension on the capillaries dominates in the neck-down region [100]. Yet, the non-Newtonian behavior of 3D printing filament and limited space for expansion restricted us from overshooting the capillary pressure [70].

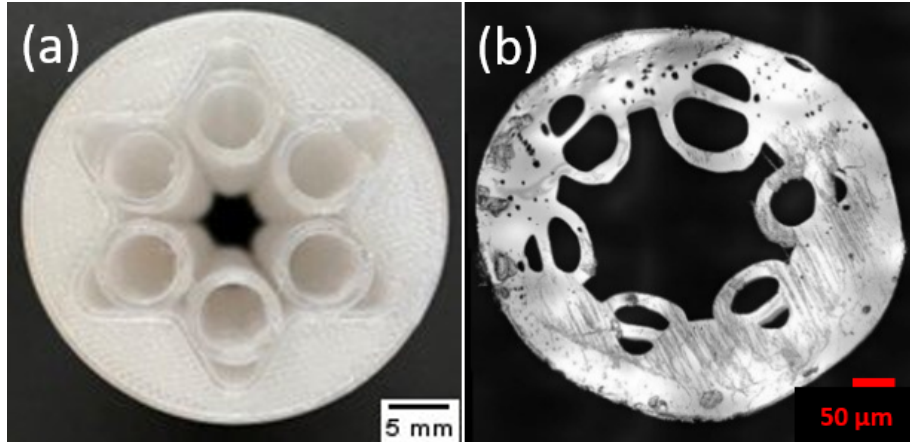


Figure 4.2: A comparison of the cross-sectional images of a) 3D printed preform and b) HCMPOF after thermal drawing.

Therefore, the fiber was drawn with high drawing tension (120 g) with optimum core-capillary gas pressure difference (10–20 mBar) to preserve the desired geometry properly. Figure 4.2. shows a comparison of the cross-sectional images between the 3D printed preform and the HCPOF after the thermal drawing process taken by a laser scanning confocal microscope (Keyence VKX 100, Japan). The HCPOF comprises $172.75 \pm 14 \mu\text{m}$ hollow core, $18 \pm 2 \mu\text{m}$ strut thickness, and $480 \pm 10 \mu\text{m}$ outer diameter.



Figure 4.3: Collected thermally drawn HCMPOF ($\sim 10\text{m}$) with preform ends.

Despite applying gas pressure through the capillaries, slight deformation of the HCPOF's cross-section was observed with non-uniform strut thickness and tube diameters. One reason behind the non-uniformity could be the expansion of the trapped air bubbles inside the 3D printed preform during the drawing process [6, 9, 10, 11]. Furthermore, expansion of these air bubbles with incomplete polymerization during 3D printing limits the drawn fiber length (10 m) due to sudden preform breakage as shown in figure 4.3.

4.3 Propagation Loss Measurement

To evaluate the light guidance properties of the drawn fiber in the NIR, the light was coupled into the hollow core of 57.8 cm long fiber by using the characterization setup depicted in Figure 2.7. The large size of the core with speckles observed from the images significantly proves the multimode nature of the fiber as shown in Figure 4.4. To confirm the light guidance through the hollow core, transmission spectra at several fiber lengths (57.8, 41.3, 31.3, and 17 cm) were collected by an OSA. The light was also intentionally coupled to the cladding to determine the guidance characteristic of the drawn polymer. Since the Z-Glass polymer has a low glass transition temperature ($T_g = 78.06^\circ \text{C}$), the fiber tip could be burned or deformed during light coupling to the cladding region [13].

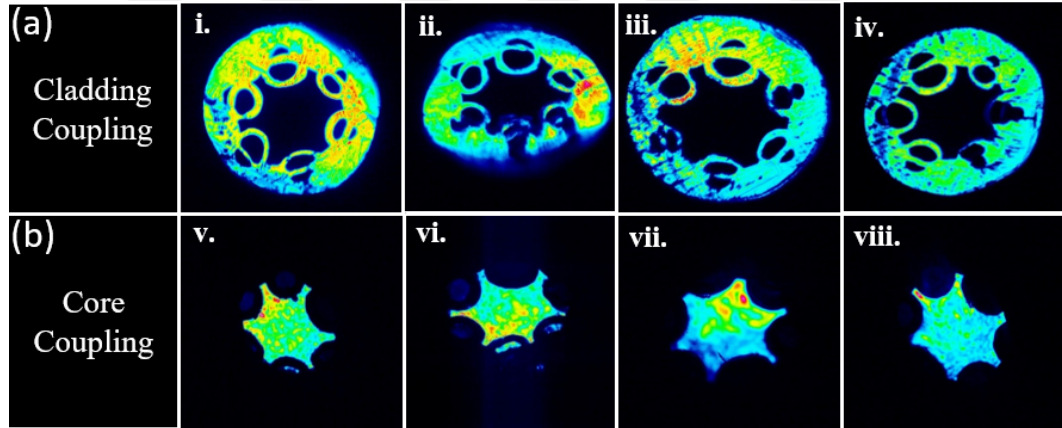


Figure 4.4: Output images of the hollow core microstructured polymer optical fiber (HCMPOF) when light is coupled to cladding (a) i - iv and core (b) v - viii at 17 cm, 31.3 cm, 41.3 cm, and 57.8 cm length, respectively.

To avoid damaging the fibers, the light source set to low power was used for straight transmission loss measurement of the fiber. Though cladding coupling at 57.8 cm long fiber can be observed from the camera, due to the material absorption transmission spectrum at this length are too noisy to be used in calculations. Furthermore, light guidance through an untreated Z-Glass filament (30 cm long filament with 1.75 mm diameter) was also observed to identify the characteristics of the untreated polymer. Figure 4.5 shows the transmission signal differences

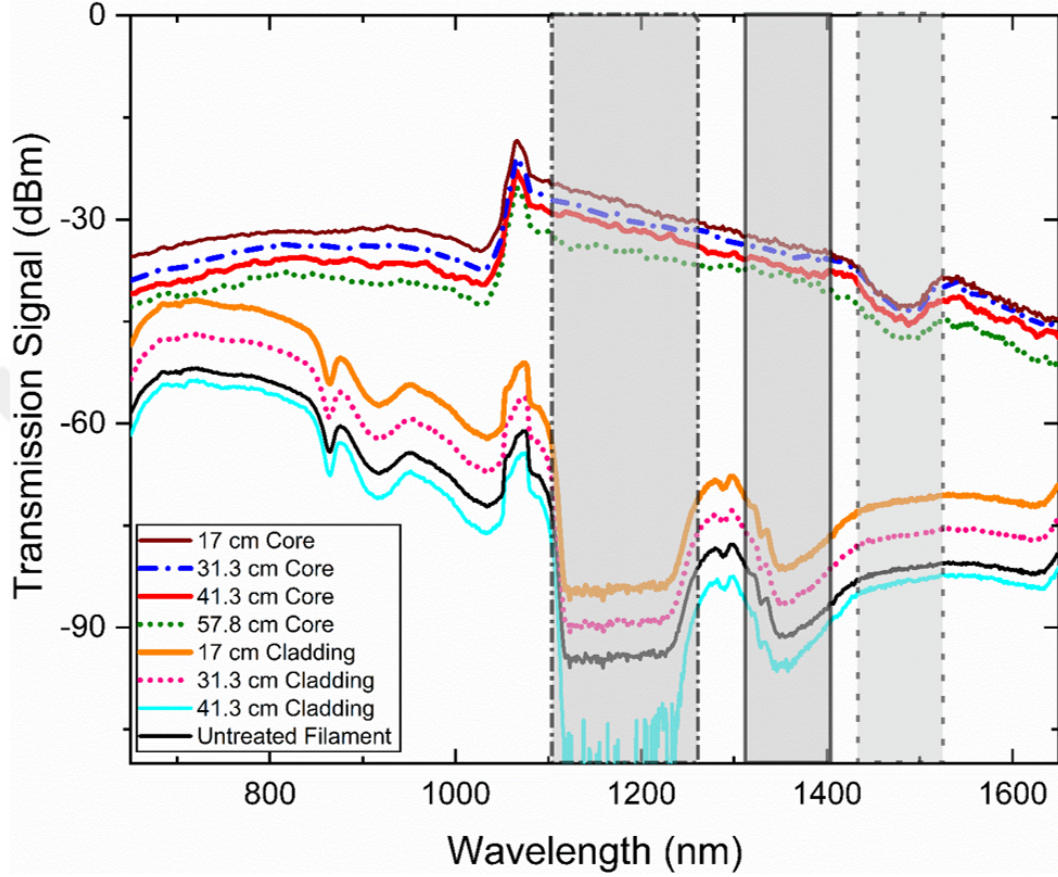


Figure 4.5: Transmission spectra of the drawn fiber at different lengths. An untreated polymer filament is also included for a better comparison.

between the core and cladding of fibers with the aforementioned lengths and untreated polymer filament. Although the transmission spectra for the fiber's core, cladding, and filament follow a similar trend between $0.65 \mu\text{m}$ and $1.05 \mu\text{m}$, where the bulk polymer is semi-transparent, a noticeable shift (grey-shaded regions) occurs at longer wavelengths due to the material absorption. The transmission behavior of the fibers in these areas supports that light is guided through the hollow core between $1.1 \mu\text{m}$ and $1.65 \mu\text{m}$. The cut-back method was used for the propagation and bending loss measurements. Since the laser source is pumped at $1.064 \mu\text{m}$ wavelength, the high loss comes from there despite high transmission signals being visible at that regime. The lowest propagation loss of 0.087 dB/cm was achieved at $1.35 \mu\text{m}$ with an average optical loss of 0.11 dB/cm between $1.3 \mu\text{m}$ to $1.4 \mu\text{m}$. Furthermore, the loss between $1.1 \mu\text{m}$ to $1.65 \mu\text{m}$

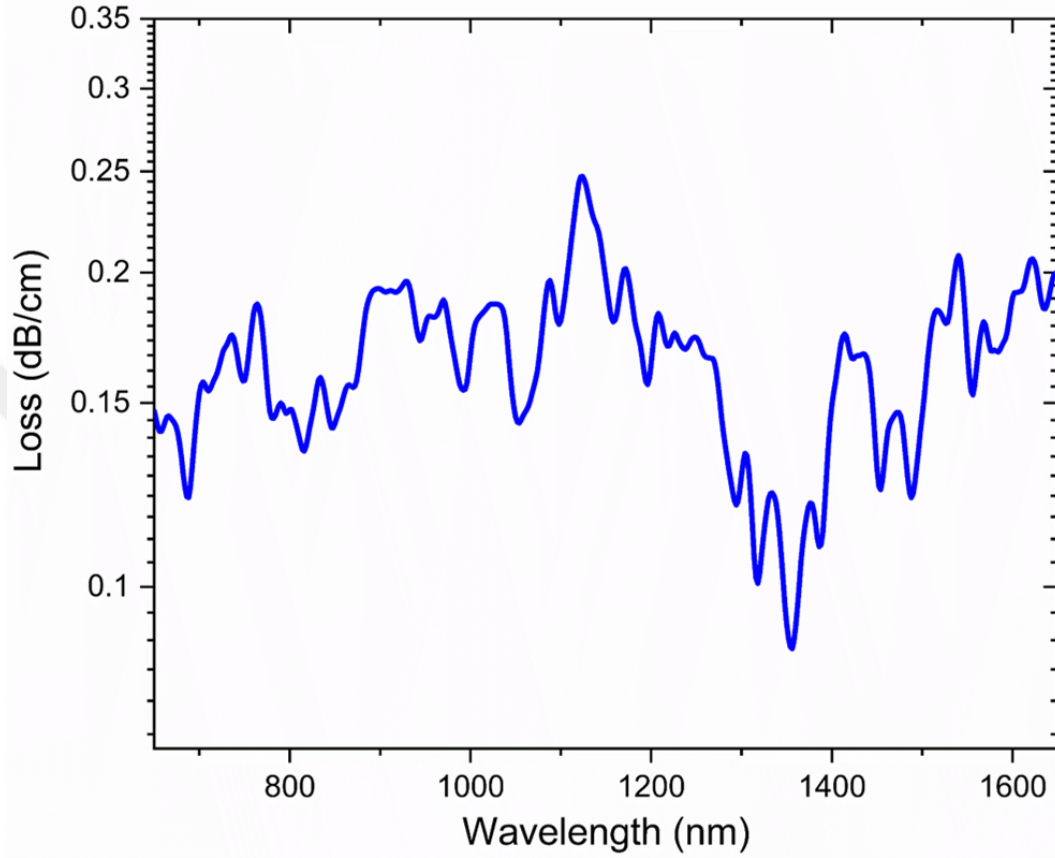


Figure 4.6: Measured propagation losses of HCMPOF between 650 nm to 1650 nm.

was averagely found as 0.16 dB/cm (Figure 4.6). Due to thick cladding elements, the transmission window is overpopulated with resonance wavelengths leading to relatively higher losses.

The fibers were further examined for mid-infrared guidance using a thermal detector (Thorlabs PM100D) and Germanium filter that only allows light guidance above $1.9 \mu\text{m}$. Using the same SC laser source, 0.23 mW power was collected, suggesting a high probability of guiding the mid-IR radiation with the proposed fiber. Further analysis with a mid-IR spectrometer would confirm the guidance by providing a detailed loss profile of the fiber at different wavelengths.

4.4 Bend Loss Measurement

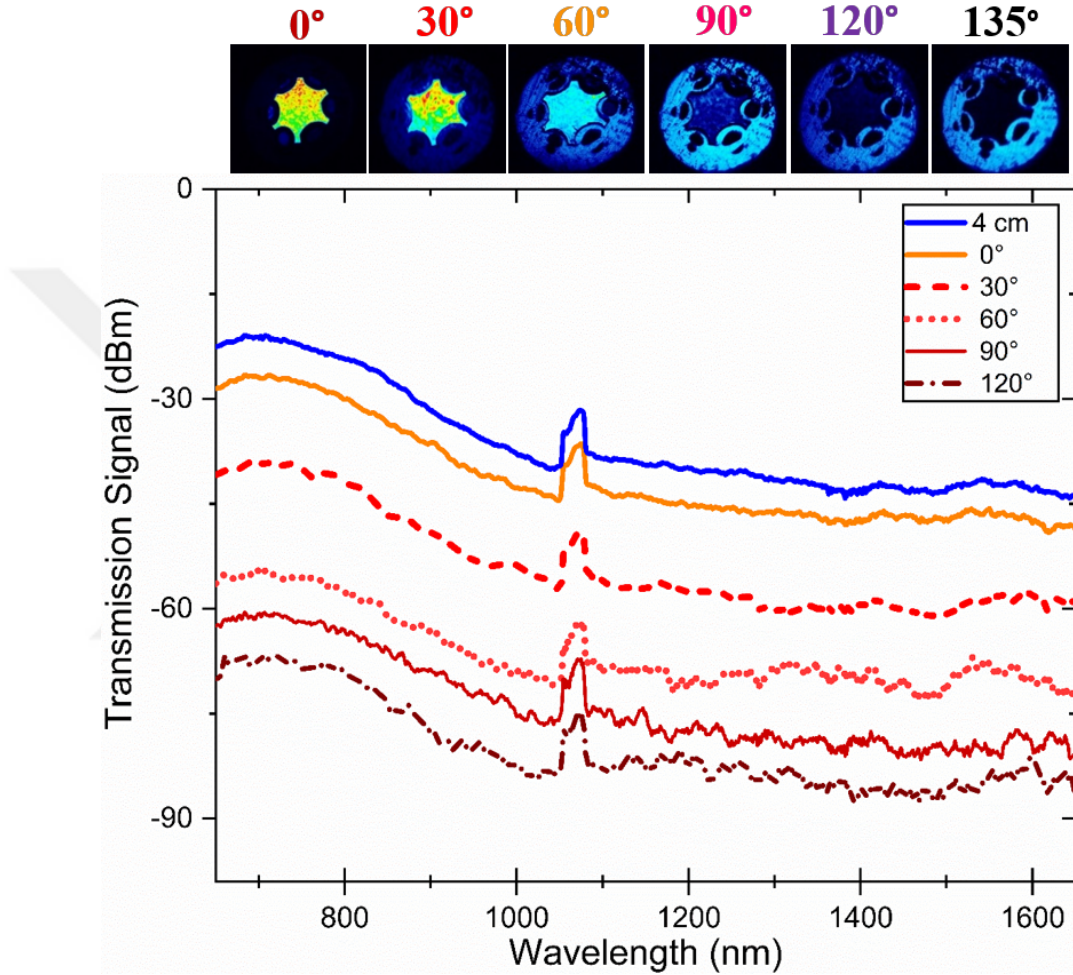


Figure 4.7: (a) Output images of the fiber and (b) Transmission signal spectra at different bending angles for 50 mm bending radius.

Bend loss measurements were performed with a different fiber collected from the same batch at 50 mm bending radius by varying the fiber angles. The light was coupled into the HCMPOF using a modified optical setup, as shown in Figure 2.8. The output images of the 31.2 cm long fiber at different angles were taken by the camera (Figure 4.7 (a)). Since the light was only coupled to the core region, the higher laser power was used for the bend measurement compared to the straight loss measurement. Hence, different transmission spectra were observed for the same fiber length collected from the same batch during propagation and bend loss measurements. At a 50 mm bend radius, transmission spectra of the core

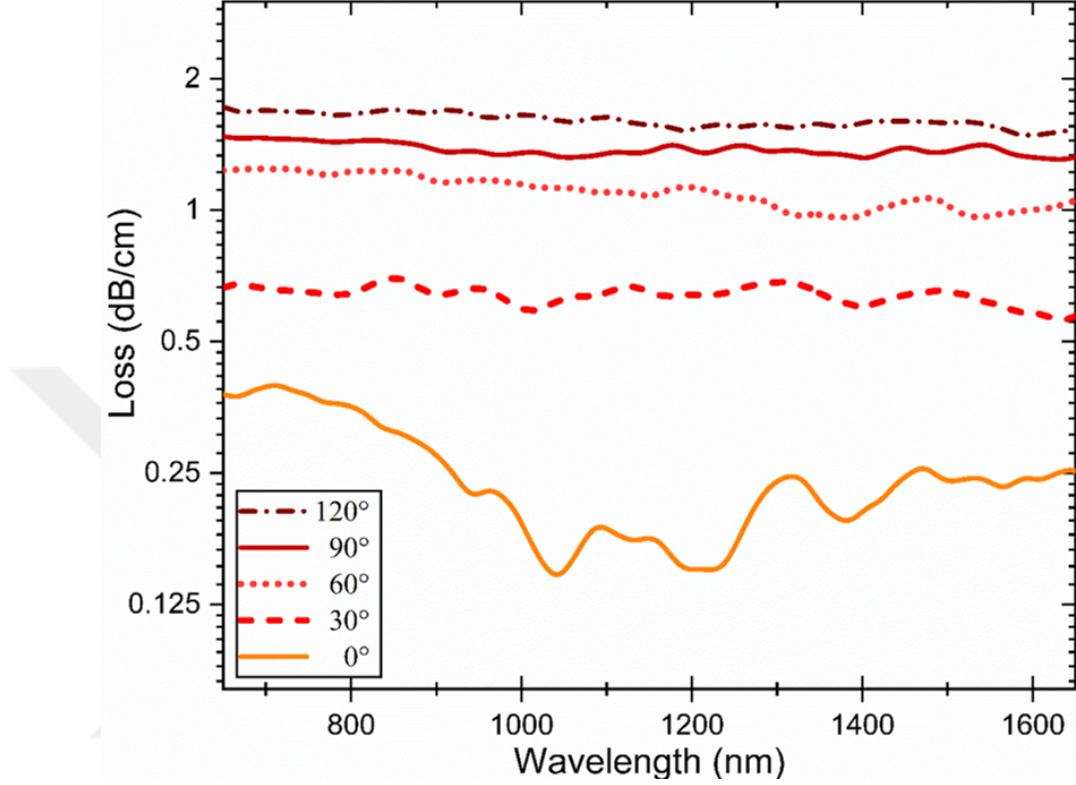


Figure 4.8: Bend induced losses of the HCMPOF for the core guidance in different angles at 50 mm bend radius.

guidance from 0° to 120° degree bending exhibit a similar profile with 31.2 and 4 cm fiber lengths (Figure 4.7 (b)). The light intensity through the core guiding decreases as the bending angle increases, resulting in cladding guidance at 135° degree bending. At $1.3 \mu\text{m}$ and $1.55 \mu\text{m}$ wavelengths, the observed bending loss for a 50 mm bend radius at 120° of bending was 1.5 dB/cm (Figure 4.8). These substantial losses can be attributed to the thicker struts and modest capillary distortion caused by trapped air bubbles' expansion during the fiber drawing. It can be further reduced by circumventing the resolution and layer thickness restriction imposed by 3D printers and minimizing the formation of trapped air bubbles during printing.

Chapter 5

Conclusions and Future Work

Light can travel 47% faster in HCF than in conventional SCOFs with lower latency, high damage threshold, and better modal properties. These properties in HCFs come with significant attention in several applications, such as telecommunication, sensing, high-power laser delivery, and non-linear optics. The transmission loss in HCFs arising from a lack of proper confinement can be reduced further by engineering the cladding structure surrounding the fiber's hollow core, such as NC-HCFs. Although silica is a material of choice for low-loss long-length guidance in the NIR, several properties, such as flexibility, durability, and bending, limit its potential use for short-distance high-speed data transmission systems. On the other hand, POFs are far more suitable due to their flexibility, easier material processing, temperature sensitivity, accessibility, and disposability. Despite higher NIR absorption of polymers than silica counterparts in NIR, the possibility of light guidance through the hollow core paves the way for achieving low transmission loss in POFs. Even though various methods for fabricating POFs from preform, such as drilling, billet extrusion, cast, polymerization, stack and draw, and 3D printing, have already been investigated, successful fabrication and optical characterization of negative curvature and intricate geometry-based HCMPOFs are yet to be resolved.

Fabrication of five tubes and six tubes based tubular NC-HCMPOFs were investigated based on the stack and draw technique with the potential to achieve long fiber length. The primary fabrication process of the preforms was systematically optimized to achieve well-structured geometry after the fiber drawing process. During the thermal drawing process, the expansion and contraction of the fiber core and cladding were frequently observed. The optimized gas pressure difference between the core and cladding parts preserved well-structured fiber geometry. The five-tube- and six-tube-based HCMPOFs consisted of core diameters of $153.1 \pm 8 \mu\text{m}$ and $68.53 \pm 5 \mu\text{m}$ and capillary diameters of $74.5 \pm 3 \mu\text{m}$ and $68.53 \pm 5 \mu\text{m}$ on average, respectively. The thickness of the capillaries surrounding the core was $4.5 \pm 0.3 \mu\text{m}$ for five tubes HCMPOF and $6.34 \pm 0.5 \mu\text{m}$ for six tubes HCMPOF. Optical propagation loss measurement was carried out with the cutback measurement approach. For the five-tube design, the lowest propagation loss (32.81 dB/m) was achieved at 670 nm wavelength with an average optical loss of 51.78 dB/m between 6.5 μm to 1 μm and 58.95 dB/m between 1.1 μm to 1.50 μm .

On the other hand, the six-tube-based design's lowest propagation loss was 25.18 dB/m at 1430 nm. This fiber possesses an average optical loss of 48.57 dB/m and 49.40 dB/m over 6.5 μm to 1 μm and 1.1 μm to 1.65 μm wavelength range, respectively. The high transmission loss in these HCMPOFs was visible due to the cladding structure's lack of proper confinement of the fibers' core. Further investigation was carried out to observe the inside geometry of the 104 cm long six tubes HCMPOF. A continuous slight change in the shape and position of the cladding parts inside the fiber was found even at a 15 cm distance. Failure of proper preform stacking could be one of the primary reasons behind the high losses. Since thinner polymer capillaries lack rigidity compared to silica capillaries, these polymer capillaries easily bend even at shorter lengths, which results in improper preform stacking. However, stacking the main capillaries tightly inside the outer jacket of the preform with the help of glass capillaries and etching those capillaries after proper stacking can be a pragmatic solution for the preform preparation. This way, we can have similar fiber geometry inside a preform, requiring less gas pressurization inside the capillaries during the

fiber drawing process. Alternatively, a larger preform size with an equal draw-down ratio can also provide well-structured fiber geometry to reduce the losses with possible resonance bandwidth in the NIR regime. Again, the fabrication and optical characterization of a novel negative curvature HCPOF from a 3D printed preform was demonstrated using low-cost and commercially available 3D printed filaments. The direct thermal fiber drawing strategy while applying the gas pressure inside the preform is less time-consuming, less labor-intensive, and better to preserve the intricate structure of the fiber. However, limitations of the 3D printing parameters, such as printer resolution and layer thickness, still have significant effects on the final fiber geometry. Light is successfully guided through the air core of 57.8 cm long fiber with low propagation losses (8.7 dB/m at 1.35 μm and an average of 16 dB/m in the observed spectrum of 650 nm to 1650 nm). Light guidance is also observed at different bending angles with a maximum loss of 1.5 dB/cm at a 50 mm bend radius at a 120° bending angle. Moreover, the average optical loss of 0.16 dB/cm between 1.1 μm to 1.65 μm is two orders of magnitude lower than the material absorption loss [10]. The losses can be further reduced using a preform printed from a high-resolution 3D printer. Future work can be focused on fabricating other intricate geometry-based HCMPOFs [58, 101] can be further explored for potential light guidance in the NIR based on additively manufactured preforms using a higher resolution 3D printer. Scaling down the layer thickness barrier in the 3D printer resonance bandwidth can also be achievable with low optical propagation loss in the NIR regime.

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