

PUBLISHED VERSION

Ebendorff-Heidepriem, Heike; Monro, Tanya Mary.

Extrusion of complex preforms for microstructured optical fibers, *Optics Express*, 2007; 15 (23):15086-15092.

Copyright © 2007 Optical Society of America

PERMISSIONS

http://www.opticsinfobase.org/submit/review/copyright_permissions.cfm#posting

This paper was published in *Optics Express* and is made available as an electronic reprint with the permission of OSA. The paper can be found at the following URL on the OSA website:

<http://www.opticsinfobase.org/abstract.cfm?URI=oe-15-23-15086>. Systematic or multiple reproduction or distribution to multiple locations via electronic or other means is prohibited and is subject to penalties under law.

OSA grants to the Author(s) (or their employers, in the case of works made for hire) the following rights:

(b) The right to post and update his or her Work on any internet site (other than the Author(s') personal web home page) provided that the following conditions are met: (i) access to the server does not depend on payment for access, subscription or membership fees; and (ii) any such posting made or updated after acceptance of the Work for publication includes and prominently displays the correct bibliographic data and an OSA copyright notice (e.g. "© 2009 The Optical Society").

17th December 2010

<http://hdl.handle.net/2440/43939>

Extrusion of complex preforms for microstructured optical fibers

Heike Ebendorff-Heidepriem and Tanya M. Monro

Centre of Expertise in Photonics, School of Chemistry & Physics, University of Adelaide, SA 5005, Australia
heike.ebendorff@adelaide.edu.au

Abstract: We report a significant advance in preform extrusion and die design, which has allowed for the first time the fabrication of complex structured preforms using soft glass and polymer billets. Structural preform distortions are minimized by adjustment of the material flow within the die. The low propagation loss of an extruded complex bismuth glass fiber demonstrates the potential of this advanced extrusion technique for the fabrication of novel soft glass and polymer microstructured fiber designs.

©2007 Optical Society of America

OCIS codes: (060.2270) Fiber characterization; (060.2280) Fiber design and fabrication; (060.2290) Fiber materials.

References and links

1. T. M. Monro and H. Ebendorff-Heidepriem, "Progress in microstructured optical fibers," *Annu. Rev. Mater. Res.* **36**, 467-495 (2006).
 2. M. C. J. Large, S. Ponratham, A. Argyros, I. Bassett, N. S. Punjari, F. Cox, G. W. Barton, and M. A. von Eijkelenborg, "Microstructured polymer optical fibers: New opportunities and challenges," *Mol. Cryst. Liq. Cryst.* **446**, 219-231 (2006).
 3. Y. Zhang, K. Li, L. Wang, L. Ren, W. Zhao, R. Miao, M. J. C. Large, and M. A. van Eijkelenborg, "Casting preforms for microstructured polymer optical fiber fabrication," *Opt. Express* **14**, 5541-5547 (2006).
 4. Z. Guiyao, H. Zhiyon, L. Shuguang, and H. Lantian, "Fabrication of glass photonic crystal fibers with a die-cast process," *Appl. Opt.* **45**, 4433-4436 (2006).
 5. P. Petropoulos, T. M. Monro, H. Ebendorff-Heidepriem, K. Frampton, R. C. Moore, and D. J. Richardson, "Highly nonlinear and anomalously dispersive lead silicate glass holey fibers," *Opt. Express* **11**, 3568-3573 (2003).
 6. H. Ebendorff-Heidepriem, P. Petropoulos, S. Asimakis, V. Finazzi, R. C. Moore, K. Frampton, D. J. Richardson, and T. M. Monro, "Bismuth glass holey fibers with high nonlinearity," *Opt. Express* **12**, 5082-5087 (2004).
 7. X. Feng, T. M. Monro, V. Finazzi, R. C. Moore, K. Frampton, P. Petropoulos, and D. J. Richardson, "Extruded single-mode, high-nonlinearity tellurite glass holey fiber," *Electron. Lett.* **41**, 835-837 (2005).
 8. J. Y. Y. Leong, P. Petropoulos, J. H. V. Price, H. Ebendorff-Heidepriem, S. Asimakis, R. C. Moore, K. E. Frampton, V. Finazzi, X. Feng, T. M. Monro, and D. J. Richardson, "High-nonlinearity dispersion-shifted lead-silicate holey fibers for efficient 1- μ m pumped supercontinuum generation," *J. Lightwave Technol.* **24**, 183-190 (2006).
 9. H. Ebendorff-Heidepriem, T. M. Monro, M. A. van Eijkelenborg, and M. C. J. Large, "Extruded high-NA microstructured polymer optical fiber," *Opt. Commun.* **273**, 133-137 (2007).
 10. E. Roeder, "Flow behaviour of glass during extrusion," *J. Non. Cryst. Solids* **7**, 203-220 (1972).
 11. A. Argyros, M. A. van Eijkelenborg, M. C. J. Large, and I. M. Bassett, "Hollow-core microstructured polymer optical fiber," *Opt. Lett.* **31**, 172-174 (2006).
 12. H. Ebendorff-Heidepriem, Y. Li, and T. M. Monro, "Reduced loss in extruded microstructured optical fiber," *Proc. Australian Conference on Fibre Technology, Melbourne (Australia)*, paper P20 (2007).
-

1. Introduction

Microstructured optical fibers (MOFs) contain an array of wavelength-scale air holes within the fiber cross-section, which allows a broad range of novel optical properties. In the last decade, silica-based MOFs have received significant attention, with some variants are now reaching maturity [1]. There is growing interest in soft glass and polymer MOFs, which significantly broaden the range of potential fiber properties [1, 2].

The successful realization of a range of MOFs depends on the use of complex air/glass structures within the fiber cross-section. Examples include air-core photonic bandgap fibers, large mode area fibers and fibers with broadband flattened dispersion, whose optical properties cannot be realized with conventional core-clad fiber designs or even simpler MOF designs. These fiber types require the fabrication of preforms with a macroscopic structure containing large numbers of transverse features. Although to date these fiber types have all been demonstrated in silica-based MOFs using the stacking fabrication technique, the development of soft glass MOFs variants has been hampered to date by fabrication challenges.

Stacking, drilling and casting techniques have been used to fabricate structured preforms. These techniques all have limitations in the number of transverse features, hole shapes and configurations that can be achieved. Stacking is essentially limited to circular holes arranged in a hexagonal lattice and is time-consuming and laborious. Drilling is limited to circular holes, and to shorter preform lengths and, for glass, necessitates preform polishing to reduce surface roughness and contamination [1]. To date, casting of preforms with large numbers of holes has only been demonstrated for circular holes using in-situ polymerization [3] and a combination of suction casting and mould rod etching [4].

A promising alternative technique is billet extrusion, which has been shown to be a versatile, reproducible single-step approach to fabrication of soft glass and polymer preforms with up to 12 holes [5-9]. However, to produce even these relatively simple structures, extrusion dies with relatively complex internal structures were used. It is not readily apparent how these die designs can be adapted and scaled to produce complex preforms. The existence of preform structure distortions relative to the die exit geometry [9, 10] also mandates careful optimization and control of not only the die design but also the extrusion conditions.

In this paper, we present a new die design concept along with advances in the extrusion process control, which together overcome these issues to allow the first demonstration of truly complex extruded glass preforms. The flow of material within the die is explored for a range of structures and materials, and the efficacy of the extruded preforms for low-loss fiber fabrication is demonstrated.

2. Advance in extrusion capability and die design

Preforms are extruded by heating bulk billets of optical materials up to a temperature where the material is sufficiently soft to allow it to be forced through a die structure by a ram into free-space to form an extrudate with a complex transverse profile [Fig. 1(a)]. In the extrusion process, both the billet temperature and ram speed are fixed, and the ram force is adjusted via feedback control to maintain a fixed ram speed. We have used three different materials for the results presented here: SF57 lead silicate glass from Schott Glass Co., a bismuth glass from Asahi Glass Co., and commercially available polymethylmethacrylate (PMMA) polymer. The extrusion temperatures for the lead silicate glass were 520-525 °C, for the bismuth glass 490-495 °C and for the polymer 170-180 °C. The extrusion speeds and pressures for the lead silicate and bismuth glass were in the range of 0.07-0.10 mm/min and 20-30 MPa, and for the polymer in the range of 0.03-0.10 mm/min and 1-7 MPa. Our extrusion apparatus allows accurate control of ram force, ram speed and billet temperature over a wide range (10N-100 kN, 0.001-200 mm/min), which has allowed us to extend the billet extrusion technique to polymer [9], to exclude sources of human error via the automation of the extrusion process and to achieve high degree of reproducibility between preforms. We have also scaled up the billet and die diameter from 30 mm (as used previously) to 50 mm, which allows considerably larger number of transverse die features without reducing the feature size to unmanageable dimensions (<1 mm diameter) in terms of die machinability and flow restrictions [10].

Our new die design concept [Fig. 1(b)] enables the extrusion of complex preforms in a single step. The extrusion die has two principal elements: holes that are used to feed material through the die ('feed holes', shown by black or red circles) and elements that block the flow of material (white shapes). Within the die, the material strands emerging from the feed holes

fuse together to form a single body, while the blocking elements obstruct the flow and thus form the holes in the preform. The extrusion speed has to be sufficiently slow to ensure that the material strands linger for adequate time within the die and thus can fuse together completely. Note that the extrusion speeds used to extrude our preforms are few orders of magnitude smaller than the speeds generally used for aluminum and polymer extrusion. Our new die design allows great flexibility in the selection of the size, shape and distribution of the feed holes, which provides control of the material flow through the die in a manner that is truly scalable, reconfigurable, easily understood and thus optimized. The die design also offers independent control of the hole shape and configuration within a preform for the first time.

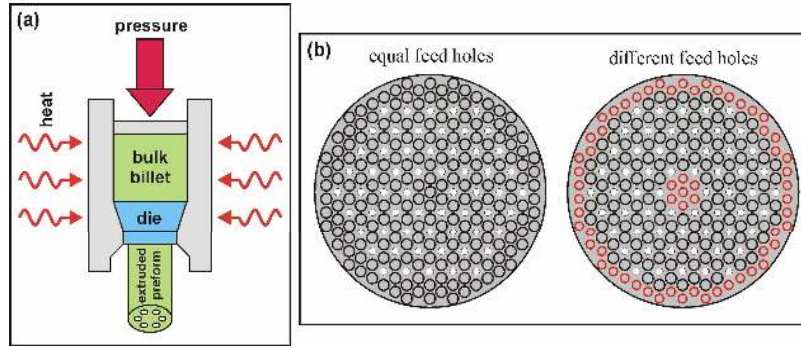


Fig. 1. (a). Sketch of extrusion process and (b) extrusion die concepts with equal and different size feed holes for a target preform structure having 60 holes (4 rings), white filled circles are blocking elements, black and red circles are feed holes.

3. Structure of complex preforms

Initially we tested the new die design for a variety of dies comprising a number of circular blockages arranged on a hexagonal lattice, i.e. targeting preform structures with hexagonal arrays of circular air holes (Fig. 2). Preforms with 36 holes (3 rings) were extruded using billets of 30 mm in diameter. To explore the potential for scaling the die design (and thus the number of holes) within an extruded preform, we increased the number of transverse features to 162 holes (7 rings) using the same size and pitch for the blocking elements in the die. The billet and die size were thus increased to 50 mm. For both the 3-ring and the 7-ring preforms and for all three billet materials, the die exit structure is well retained (Fig. 3).

Closer inspection of the transverse structure within the extruded preforms shows that some of the holes are slightly deformed and/or dislocated relative to the die exit geometry. In order to gain more insight into these preform structure distortions, we measured the position and shape of the holes in the glass preforms from cross-sectional images as follows. Holes arranged on a hexagonal lattice around a solid core can be classified according to their distance from the core (Fig. 2), where Λ is the hole-to-hole pitch, d is the hole diameter, N refers to the relative position of a hole from the preform centre (i.e. ring number counted from the core), and n refers to the position of a hole within a ring relative to the nearest corner hole. The shape of each hole was approximated by an ellipse. From the position of the ellipses, the distance, r , of the centre of each ellipse relative to the centre of the structure was calculated. The values for the short and long axis and the distance of the ellipses were averaged for each hole type. The hole-to-hole pitch of each hole type was calculated via

$\Lambda = r / \sqrt{N^2 - N \cdot n + n^2}$. The elliptical hole shape is given by the ratio of long to short axis of the ellipses. The scale of the structures was normalized by using the pitch of a corner hole type, which shows the least structure distortions (i.e. '2a' holes for 3- and 4-ring structures, '3a' holes for 7-ring structure), as the reference pitch Λ_{ref} . The relative hole position of a hole type is then defined as the ratio of the pitch of this hole type to the reference pitch, $\Lambda/\Lambda_{\text{ref}}$. For a cross-sectional image of a preform, the standard deviation within the measurements of the

individual holes of a given hole type is in the range of 0-6% for the hole shape and 0.2-1.3% for the hole position, which implies that a high degree of similarity in the location and shape of the individual holes of a given hole type was achieved. The magnitude of this error is determined by the quality of both the preform cross section and its image. For two images taken from the same preform, the deviations in the average values for position and shape of a hole type are smaller than the deviations between the individual holes of a hole type.

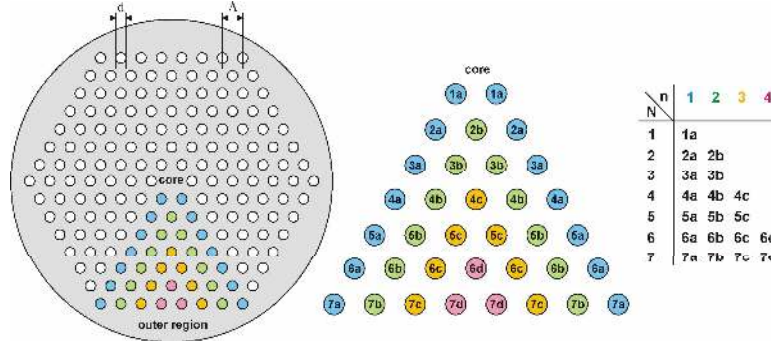


Fig. 2. Hole type classification and labeling for transverse preform structures containing air holes arranged on a hexagonal lattice.

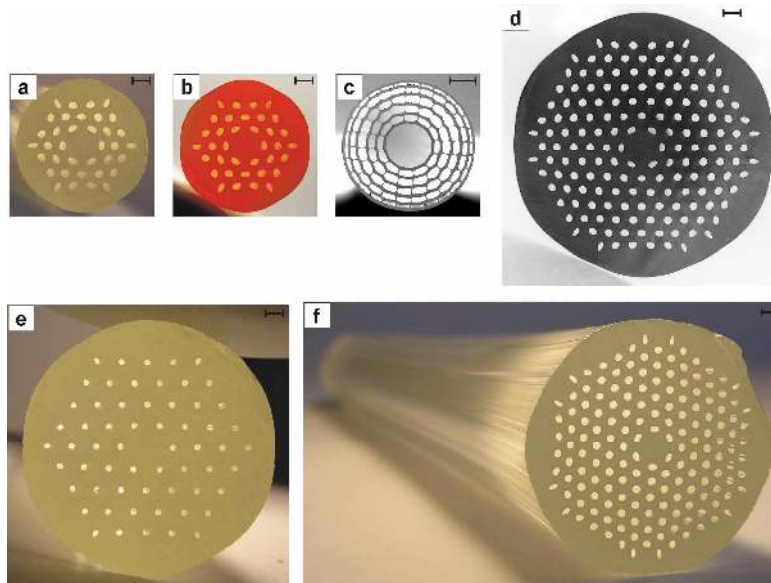


Fig. 3. Photographs of extruded preforms. (a), (e) and (f) lead silicate glass; (b) bismuth glass; (c) and (d) polymer. The bars refer to 2mm.

The measurements reveal two types of structural distortions: dislocation of the hole positions relative to that of an ideal hexagonal lattice and elliptical deformation of the hole shapes. The structural distortions observed in the hole regions adjacent to the solid core and outer regions are larger than in the other hole regions. The latter preform regions correspond to regions within the die cross-section where each blocking element is surrounded evenly with feed holes over 3 or more periods of the hexagonal lattice of blocking elements, which for example corresponds to the 3rd, 4th and 5th ring of holes ($N = 3, 4, 5$) of the 7-ring preform (Fig. 2). The negligible structural distortions in these regions (Fig. 4) demonstrate that when equal flow is achieved around each blocking element, structural distortions are minimized, and the hole configuration achieved strongly resembles those within the die exit. By contrast,

in the holey regions adjacent to the solid core and outer regions, the holes of the innermost ring are pushed outwards, whereas the holes within the outermost ring are pushed inwards relative to the position of the hexagonal lattice (as defined by the reference pitch). Likewise, the elliptical hole deformation is large for the innermost and outermost holes (Fig. 4).

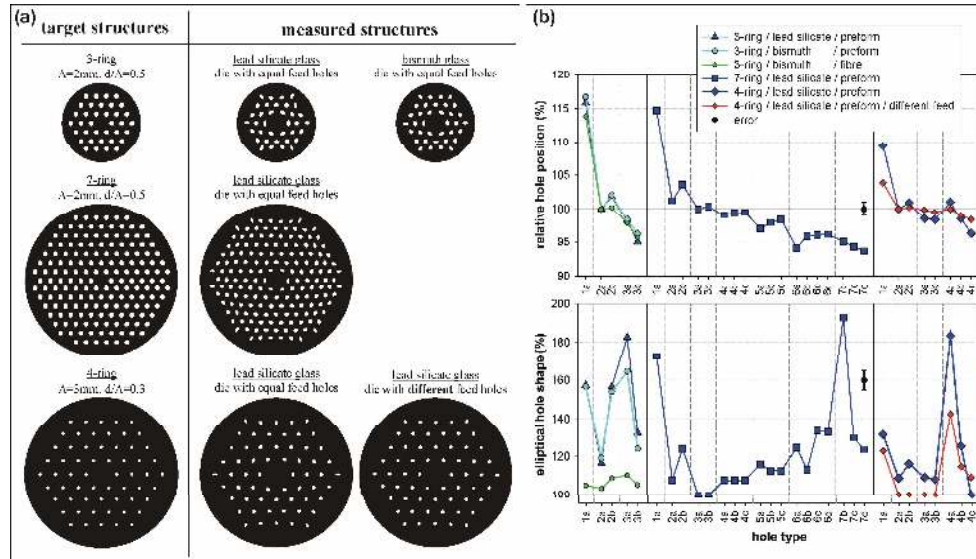


Fig. 4. (a). Transverse profiles of targeted and measured preform structures and (b) measured position and shape of holes in preforms and fiber.

To form the solid regions of a preform, it is necessary to omit blocking elements and thus to fill additional cross-sectional area within the corresponding die regions with glass. In the dies presented so far, this has been readily achieved via the incorporation of additional feed holes at the positions of the missing blocking elements. However, the hole distortions that can be observed adjacent to the solid regions demonstrate that the additional feed holes used within these examples resulted in too much glass flow in these regions. To reduce the flow, we explored the impact of reducing the feed hole size for two different die designs [Fig. 1(b)] containing 60 blocking elements (4 rings). One of these designs has constant feed hole sizes throughout (as for the initial designs described above) and one in which the feed holes in the inner and outermost regions have been reduced in size relative to the feed holes in the other regions. Considering practical issues such as die machinability and friction enhancement within small feed holes, the size of the reduced size feed holes was limited to 80% of the size of the regular feed holes. The larger distance between the blocking elements in the 4-ring die ($A=3\text{ mm}$) compared with the 7-ring die ($A=2\text{ mm}$) allows incorporation of feed holes of reduced size with manageable small dimensions. The performance of the two dies was explored using lead silicate glass under identical extrusion conditions. The smaller structure distortions in the preform extruded through the die that uses smaller feed holes shows that the reduction in feed hole size has indeed reduced flow enhancement (Fig. 4).

One distinct advantage of extrusion is that non-circular holes can be produced, which has been demonstrated to date for relatively simple preforms with 3 non-circular holes [5-9]. With our new die design concept, large numbers of non-circular holes can be readily produced by using blocking elements of appropriate shape. This was demonstrated for a polymer preform extruded through a die with 100 segment-shaped blocking elements evenly distributed in rings around the circular blockage in the centre [Fig. 3(c)]. Since only the shape of the blocking elements was modified, the same extrusion speed as for the dies described above was used. The hollow-core preform structure is of particular interest for air-core photonic bandgap fibers [11] and exemplifies how extrusion can produce geometries that cannot be achieved by

drilling and stacking.

4. Fiber fabrication and propagation loss

The suitability of our extruded preforms for fabrication of low-loss MOFs was explored for the 3-ring bismuth glass preform. This glass and structure is of interest for high-nonlinearity fibers with zero dispersion in the near-infrared [8]. The 3-ring preform was scaled down in size to a cane of 1 mm outer diameter, which was inserted into an extruded jacket tube, and finally this assembly was drawn down to the fiber. The slight overpressure within the holes during fiber drawing significantly reduced the hole shape deformation but did not remove the dislocation of the innermost holes (Figs. 4,5), which underlines the need for flow adjustment within extrusion dies. The minimum loss achieved within the 3-ring bismuth fiber is 1.2 dB/m at 1100 nm (Fig. 5), which is lower than the loss achieved with a small-core extruded MOF previously made from bismuth glass [6]. The steep increase of the loss below 800 nm is the tail of the transmission edge of bismuth glass at ~500 nm [6]. The absorption band at 1450 nm and the loss increase above 1400 nm is due to hydroxyl groups in glass [1]. The low loss of the MOF made using the new die design demonstrates that the fusion of the material strands within the die did not form interfaces within the extruded preform which would lead to enhanced loss. The low loss of the MOF described here is consistent with our recent result of negligible excess loss for the first time in a small-core extruded soft glass MOF relative to an unstructured fiber made of the same glass [12]. The advances in extrusion die design and process control described here account for the minimization of excess loss in extruded MOFs.

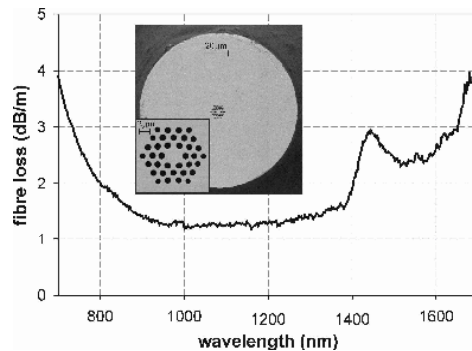


Fig. 5. Propagation loss and cross-sectional image of bismuth fiber with 3 rings of holes.

5. Summary and Conclusions

The successful extrusion of a variety of preforms with up to 162 holes using two glasses and a polymer demonstrates that the proposed new die design concept and significant advances in extrusion process control is suitable for production of preforms with large numbers of transverse features within different material types. With this new type of die design, the maximum number of holes that can be achieved in a preform is only limited by the maximum die and billet size that can be used in the extrusion apparatus. The flexibility in the die design will allow new preform structures such as non-hexagonal lattices, non-circular holes and varied hole sizes and shapes within one preform, eliminating structure deformation by feed hole size adjustment. In the future, modeling of the extrusion flow will further improve die design and optimization of extrusion conditions and thus ultimately enable the development of photonic band gap and large mode area fibers in soft glasses and polymer.

Acknowledgments

We acknowledge the DSTO (Australia) for support for the Centre of Expertise in Photonics; P. Davies at DSTO for support on die design and fabrication; Asahi Glass Co. for the fabrication of the bismuth glass billets; M.C.J. Large, M.A. van Eijkelenborg and A. Argyros

at Sydney University for collaboration on polymer preforms, W. Zhang and T. Gueder at Adelaide University for their help with the extrusion experiments, Y. Li at Adelaide University for the fiber loss measurement.