厚生労働科学研究費補助金
医療機器開発推進研究事業

極細径内視鏡用高機能中空ファイバの製作に関する研究

平成21年度　総括研究報告書

研究代表者　**岩井　克全**

平成22（2010）年３月

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研究代表者　岩井　克全　仙台高等専門学校情報ネットワーク工学科　助教

研究要旨：本研究では、①無機材料が内面にコートされた銀中空ファイバを用いる、②内径 0.1 mm の細径中空ファイバ、③製作法が単純、低コスト化が可能の特長を有する超細径中空ファイバの製作を目的とする。高エネルギー伝送ならびに滅菌工程に耐える超細径中空ファイバが実現できれば、歯科内視鏡などの低侵襲治療の高効率化を図ることが可能である。

研究分担者
　なし

A．研究目的

治療用の数多くのレーザの中、波長が 2 μm 以上の赤外波はその有効性が確認されつつも、レーザ発振器から患部へのレーザ光の導光手段は従来の石英光ファイバが使えないため、最近は図 1 のような中空ファイバが多く使用されてきている。

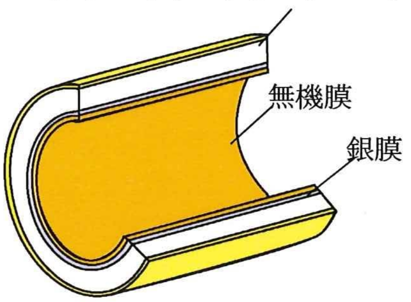

図 1 中空ファイバの構造

中空ファイバで効率的な内視鏡治療を行うためには、患部に接触して使用できること、高効率でレーザ光を導光できること、高エネルギー伝送に耐えること、内視鏡に挿入可能な細径ファイバであることが要求されている。現時点ではこの要求性能を満たす中空ファイバが無いため、導光効率を犠牲にした短尺の中実のガラスファイバ素子が使用されている。極細径内視鏡用の場合、ファイバの細径化によるレーザエネルギー密度の増加で従来の光学ポリマー膜の損傷が問題となるが、最近、耐久性に極めて優れた無機ガラス材料を用いた中空ファイバ（内径 0.7 mm、長さ 10 cm)の試作に成功した。無機ガラス薄膜を内装した超細径中空ファイバが実現されれば、その導光効率の高さ、高耐久性のメリットで、歯科内視鏡（図 2, 3 参照)などの低侵襲治療の高効率化を図ることが可能である。

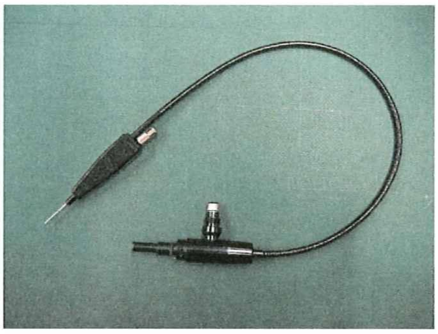

図 2 歯科内視鏡

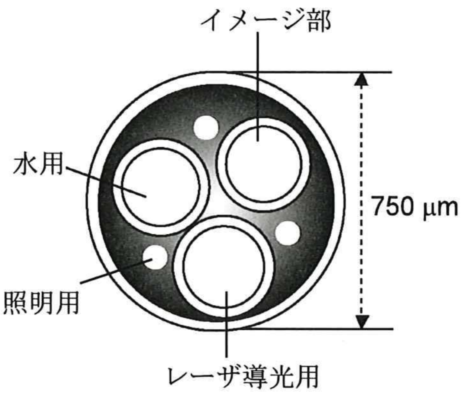

図 3 内視鏡の先端部

本研究では、図 4 のように使用可能な超細径中空ファイバとして、
①無機材料が内面にコートされた銀中空ファイバを用いる
②外径 0.17 mm、内径 0.1 mm の細径中空ファイバとして機能する

③製作法が単純で、低コスト化が可能である
の特長を有し、現存する赤外伝送路に対して、機能、価格の上で極めて優位に立つことが期待される超細径中空ファイバの製作を目的とする。

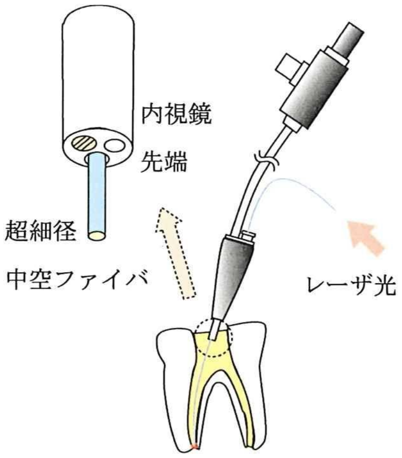

図 4 内視鏡用中空ファイバ

進捗状況並びに達成すべき課題の事業年度並びに数値レベルは、以下の通りである。

①平成 20 年度　超細径銀中空ファイバの製作
目標値：内径 0.1 mm、長さ 25 cm
達成値：内径 0.1 mm、長さ 35 cm、伝送損失 3 dB (波長 1 μm、長さ 20 cm)

②平成 21 年度　無機ガラス薄膜内装中空ファイバの製作
目標値：内径 0.1 mm、長さ 10 cm、Er:YAG 透過率 60 %

③平成 22 年度　超細径中空ファイバの先端封止技術の開発
先端封止部の Er:YAG 透過率 70%

なお、現状では、レーザ光用の先端装置で、赤外光の透過率は 30 %を越えるものはない。このことはレーザへの要求を半減できる。また、本研究で提案するような細径中空ファイバは存在しない。

技術課題は、中空ファイバの銀膜の成膜が本質的な課題であり、平成 20 年度の研究で、内径 0.1 mm、長さ 25ｃｍの超細径銀中空ファイバの製作を行い、液相法による銀膜形成条件を明らかにした。

平成 21 年度の研究では、Er:YAG レーザ（波長 2.94 μm)を高効率に伝送するために、無機ガラス薄膜内装銀中空ファイバ（内径 0.1 mm、長さ 10 cm)の製作を行い、液相法による無機ガラス薄膜形成条件を明らかにすることを目標とする。

B．研究方法

赤外レーザ光を低損失かつフレキシブルに伝送可能な伝送路として、ガラスキャピラリチューブ内面に銀層と光学膜を形成した光学膜内装銀中空ファイバがある。光学膜内装中空ファイバは、内装する光学膜の膜厚を任意に変えることで目的とするレーザ光の発振波長で低損失とすることができる。内装低損失化光学膜に要求される条件としては、

①当該波長帯で透明であること
②送液・乾燥によって一様な膜が成膜可能なこと
③耐久性に優れること

が必要である。この 3 つの条件を満足する光学膜を種々検討した結果、室温湿気硬化型特殊無機塗料である OC No. 300 クリヤー（OC300)が一つの候補であることが分った。図 5 に示すように、OC300 は主剤と硬化剤を混合することで、脱水反応により、室温で無機硬化ガラスを形成することが出来る。

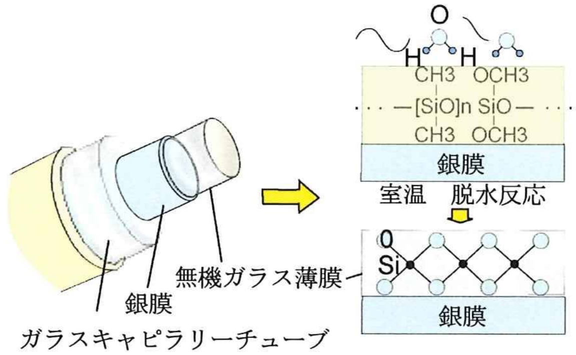

図 5 無機硬化ガラス膜（OC300)の形成

Er:YAG レーザ光用中空ファイバの伝送するモードは、市販のレーザとの結合の良いハイブリッドモードの HE_{11} モードである。中空ファイバの HE_{11} モードは TE モードと TM モードが 1:1 の割合で構成されているので、低損失に伝送させるためには光学膜境界面における TE モードと TM モードの反射係数の和を大きくするように光学膜の膜厚を設計する必要がある。図 6 に内径 0.1 mm、長さ 10 cm の銀中空ファイバの内壁に、無機ガラス薄膜を 1 層形成した OC300 膜内装銀(OC300/Ag)中空ファイバの HE_{11} モードの伝送損失を、光学膜厚の関数として示す。伝送する Er:YAG レーザ光の波長は 2.94 μm、無機ガラス薄膜（OC300)の屈折率は、1.41 としている。また銀の複素屈折率は、波長 2.94 μm で 1.26－j17.9 としている。図 6 の点線は、光学膜をコーティングしていない銀中空ファイバの損失値を示している。図 6 の結果から、無機ガラス薄膜（OC300)の最適膜厚は、0.41 μm 程度と分った。また膜厚 0.2 μm～0.6 μm 程度を成膜しても、銀中空ファイバより、低損失な無機ガラス薄膜内装銀中空ファイバが得られることが分った。

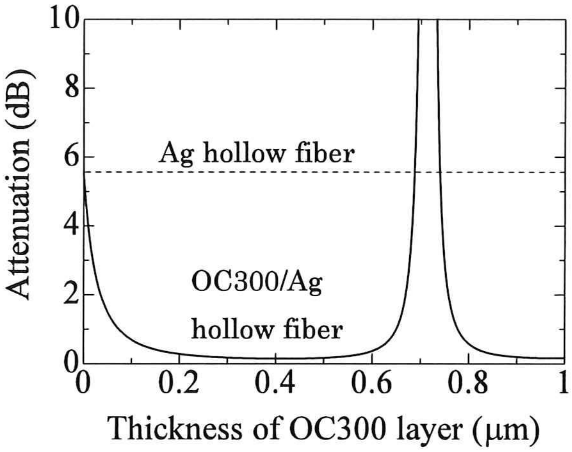

図 6 光学膜厚に対する HE_{11} モードの伝送損失
但し、内径 0.1 mm、長さ 10 cm

銀中空ファイバの内側に無機ガラス薄膜(OC300)を一様に成膜させるために送液法を用いる。送液コーティング装置を図 7 に示す。銀鏡反応によって製作した銀中空ファイバを垂直に設置する。OC300 溶液の注入用、排出用に内径 0.7 mm のガラスキャピラリーチューブを銀中空ファイバに連結させる。接続には内径 0.8 mm のタイゴンチューブを用いている。上側の溶液注入用ガラスキャピラリーチューブに OC300 溶液を約 0.03 ml 注入し、垂直に設置して、マイクロチューブポンプにより、OC300 溶液を一定速度で上から下の方向に送液を行う。

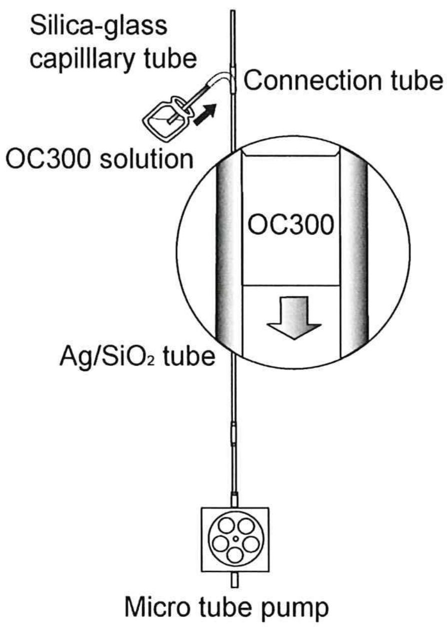

図 7 銀中空ファイバへの光学膜の送液法

送液が完了したら直ちに室温乾燥工程に移る。送液後、ファイバ内には OC300 溶液が一様に付着しているが、まだ液体状態であるために、この状態でファイバを動かすと、光学膜は変形して、ファイバの特性は悪くなる。そこで、無機ガラス薄膜をファイバに固定する目的で窒素を 100 ml/min 程度流して室温乾燥を行った。

最適膜厚を成膜するために、OC300 溶液の成膜条件について検討を行った。膜厚の制御は、
(1) 溶液の濃度
(2) 送液速度
で行うことができる。内径 0.1 mm 中空ファイバに送液を行う場合、送液速度は、一様な膜が成膜できるように可能な限り、低速度としたいため、溶液の濃度によって膜厚を制御する。

OC300 溶液は、主剤と硬化剤の割合を 10:1 とし、薄め液で濃度の調整を行った。図 8 に OC300 溶液の濃度に対する無機薄膜の膜厚を示す。内径 1 mm、内径 0.7 mm、内径 0.32 mm 中空ファイバの送液速度は、それぞれ 1.6 cm/min、4 cm/min、15 cm/min である。Er:YAG レーザ用の最適膜厚は 0.41 μm であり、濃度 32 wt%～38 wt%程度を用いるとよいと分った。

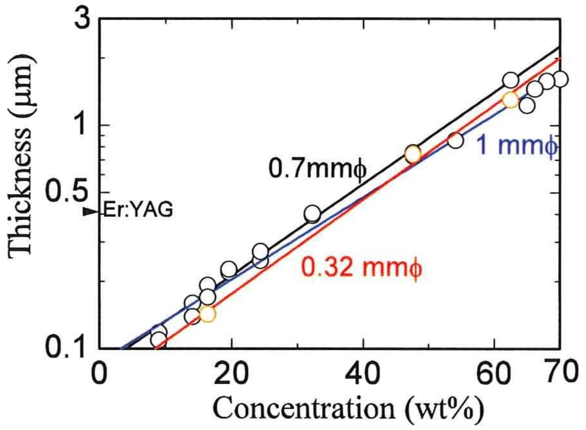

図 8 無機溶液(OC300)の濃度に対する膜厚

無機ガラス薄膜(OC300)内装銀中空ファイバの製作の際、無機ガラス薄膜の一様成膜を行うためには、OC300 溶液の送液速度を一定にすることが必要である。従来の送液系では、OC300 溶液に対して一方向から力を加えていた。これでは濃度 32 wt%程度の濃い溶液を用いると、粘度が高いため、送液速度が大きく変化する。図 9 に示すように、双方向から力を加えればより安定した速度で送液可能と思われる。そこで、安定した送液を行うために、送液系をダミーチューブで循環させ、無機溶液を上・下部から力を加える手法を用いる。図 10 に改善した OC300 溶液の送液法を示す。

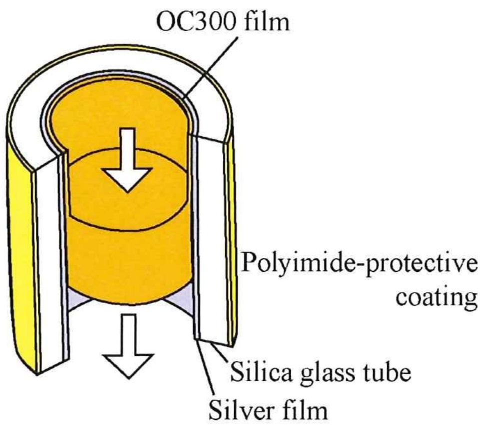

図 9　光学膜の送液法の改善

ダミーチューブで循環させることにより、送液ポンプの吸引と送り出す力を OC300 溶液へ同時に加えることが出来る。また送液の際、従来は外気を吸引していたが、改善した送液法では、蒸発した溶媒雰囲気中で送液することになるため、光学膜の乾燥に対しても有効となる。

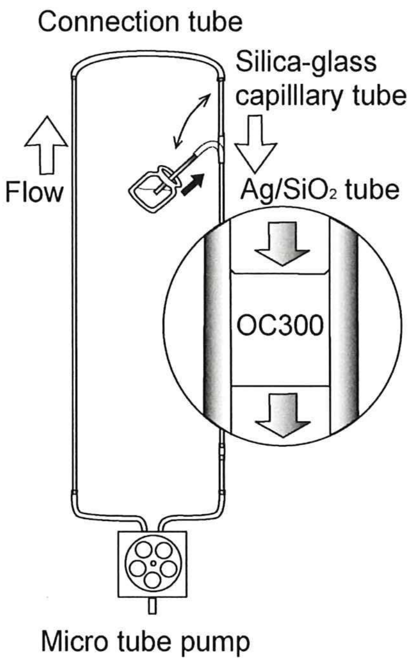

図 10 改善した OC300 溶液の送液法

中空ファイバの内径が細くなると、OC300 溶液の流速が急激に速くなる。そのため、送液系の接続点におけるチューブ径の違いにより、送液速度が変化し、ファイバの上部と下部で膜厚変動を生じると思われる。そこで、通常用いる内径 0.7 mm の接続チューブを内径 0.53 mm と 0.25 mm の 2 段階接続（図 11 参照)に改良することにより、接続

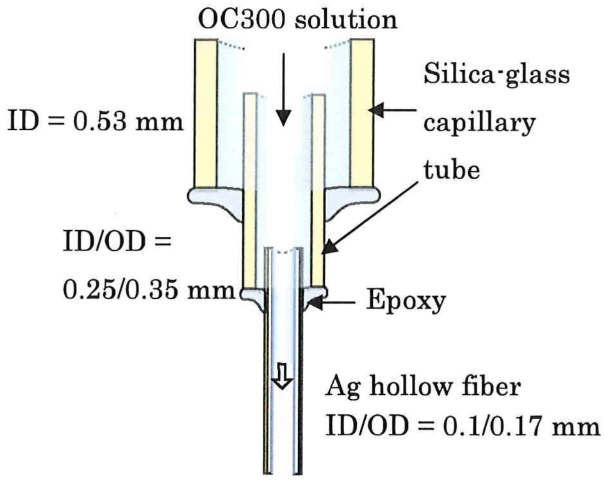

図 11 ファイバ接続点の改良

点における送液速度の変動を抑え、均一な光学膜形成を行う。

上記の送液法を用い、成膜条件は、

① 無機溶液の OC300 溶液を用いる
② 濃度は 32 wt%～38 wt%
② 送液速度は 10 cm/min
③ 送液後、室素ガスを流量 100 ml/min で流しながら、室温乾燥を 1 時間

である。送液速度は、速いほど光学膜の膜厚の変動が大きくなる傾向があるため、一様な光学膜を成膜するために、マイクロチューブポンプで送液可能な最低速度の 10 cm/min とした。室温乾燥時間は、これまでに製作した内径 0.7　mm の無機ガラス薄膜内装銀中空ファイバの製作条件から、1 時間を用いた。内径 100　μm、長さ 25 cm の銀中空ファイバに送液を行い、無機薄膜を成膜後、膜厚の変動が大きい上部と下部を切断することで、無機薄膜内装銀中空ファイバの長さは 10 cm とした。

(倫理面への配慮)

当該研究は、倫理面の問題がない。生命倫理・安全対策に対する取り組みが必要とされている研究に該当しない。

C．研究結果・考察

濃度 32 wt%の OC300 溶液を用いて、無機ガラス薄膜内装銀中空ファイバの製作を行った。光スペクトラムアナライザを用いて、無機ガラス薄膜内装銀中空ファイバ（内径 0.1 mm、長さ 10 cm）の損失波長スペクトルの測定を行った。図 12 に、無機ガラス薄膜内装銀中空ファイバの損失波長特性（FWHM10.6 ° のガウスビームで励振)を示す。

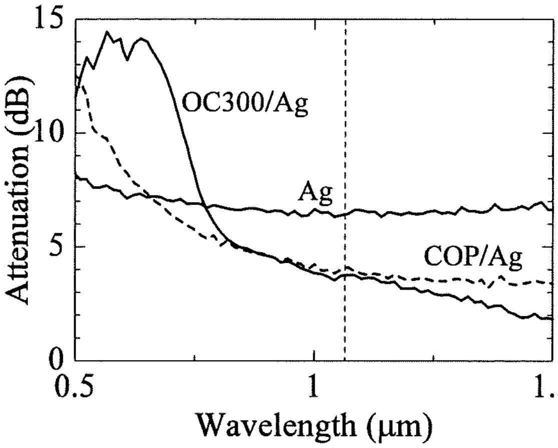

図 12　超細径無機ガラス薄膜内装銀中空ファイバの損失波長特性
但し、FWHM10.6 ° のガウスビームで励振されている。OC300 溶液の濃度は 32 wt%使用。

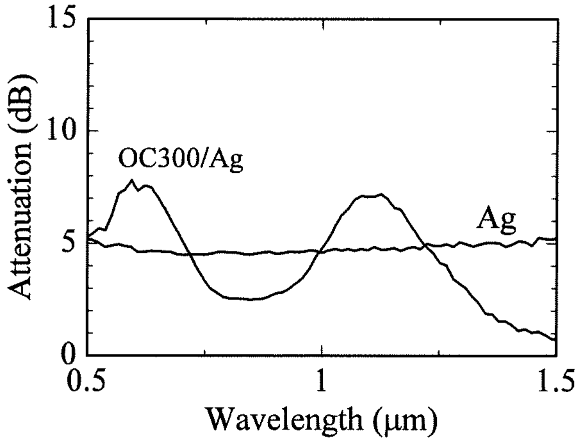

図 13　超細径無機ガラス薄膜内装銀中空ファイバの損失波長特性
但し、FWHM10.6 ° のガウスビームで励振されている。OC300 溶液の濃度は 38 wt%使用。

比較のため、従来から光学膜材料として用いられている環状オレフィンポリマー(COP)を内装した中空ファイバの損失波長スペクトルも示す。COP 溶液は濃度 8.5 wt%を用いた。OC300 内装銀中空ファイバと COP 内装銀中空ファイバは、それぞれ膜厚およそ 0.15 μm と 0.09 μm の光学膜を成膜できた。この中空ファイバは、波長 1.064 μm の Nd:YAG レーザ光用として用いることができるが、Er:YAG レーザ光用の中空ファイバは、更なる光学膜の厚膜化が求められる。溶液濃度を濃くして厚膜化を図ると、COP 溶液の粘度は高くなり過ぎ、これ以上の厚膜化は困難であった。OC300 溶液は COP 溶液と比較して粘度が低く、溶液濃度を上げて送液することが可能と思われる。

次に OC300 溶液の濃度を 38 wt%にして、送液を行った。図 13 に製作した内径 0.1 mm 無機ガラス薄膜内装銀中空ファイバ（長さ 10 cm)の可視･近赤外波長帯における損失波長スペクトル(FWHM10.6 ° のガウスビームで励振)を示す。明確な干渉ピークが見られ、銀中空ファイバ内に均一な光学膜を形成できた。無機ガラス薄膜(OC300)の膜厚は 0.27 μm である。波長 1.5 μm で 1 dB 程度と低損失な無機ガラス薄膜内装銀中空ファイバを製作できた。図 6 の計算結果から、膜厚 0.27 μm でも Er:YAG レーザ光を低損失に伝送できることが分る。

中空ファイバの評価法として、中赤外波長帯(波長 1.5 μm～12 μm)における損失波長特性を測定するために FT/IR（フーリエ変換赤外分光光度計)を用いる。光源にはインコヒーレントな特殊光源を用い、出射されるビームは半値広がり角（FWHM 12°)のガウスビームである。被測定ファイバへの結合は、内径 0.7 mm、長さ 10 cm の銀中空ファイバを結合導波路として用い、バッドカップリングで行う。ファイバから出射される光は KRS-5 レンズにより集光し検出している。この FT/IR を用いた内径 0.1 mm の無機ガラス薄膜内装銀中空ファイバの測定は困難であった。そこで、内径 0.7 mm、長さ 1 m で同程度の膜厚を有する無機ガラス薄膜内装銀(OC300/Ag)中空ファイバを製作し、FT/IR を用いて測定を行った。図 14 に内径 0.7 mm、長さ 1 m の無機ガラス薄膜内装銀中空ファイバの損失波長特性を示す。

波長 0.5 μm～1.7 μm の範囲は、光スペクトラムアナライザを用いて測定を行い、波長 1.5 μm～12 μm までは、FT/IR を用いて測定を行った。波長 3 μm 付近と 6 μm 付近に見られる損失は空気中の水の吸収によるものであり、波長 4.2 μm 付近に見られるのは空気中の二酸化炭素の吸収によりものである。波長 3.4 μm と 9 μm 付近に見られるのは OC300 の材料固有の吸収ピークである。一方、可視波長帯に見られる損失の周期的変化は干渉による損失のピークによるものである。可視波長帯における損失波長特性より膜厚を推定すると、0.32 μm であり、内径 0.1 mm 無機薄膜内装銀中空ファイバの膜厚 0.27 μm と同様に、Er:YAG の最適膜厚の 0.41 μm より少し薄いことが分る。この無機ガラス薄膜内装銀中空ファイバは、Er:YAG レーザ光の波長 2.94 μm で干渉波形の谷の部分になることから、Er:YAG レーザ光を低損失に伝送できる。

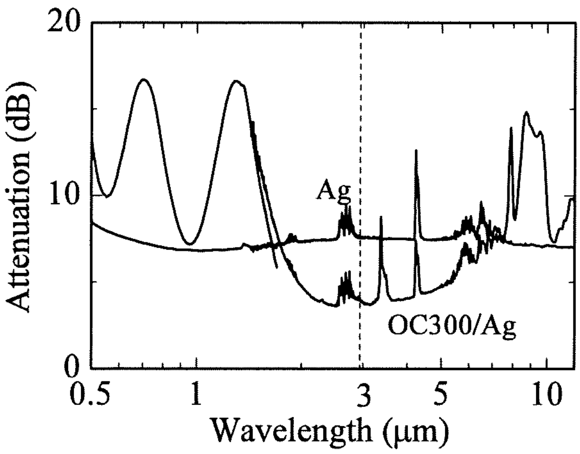

図 14　無機ガラス薄膜内装銀中空ファイバの損失波長特性
但し、内径 0.7 mm、長さ 1 m の中空ファイバを測定。波長 0.5 μm～1.7 μm まで、FWHM10.6°のガウスビームで励振され、波長 1.5 μm～12 μm まで、FWHM12°のガウスビームで励振されている。

図 14 の中赤外波長帯（波長 1.5 μm～12 μm）における損失波長特性の測定結果から、内径 0.1 mm 無機ガラス薄膜内装銀中空ファイバ（無機薄膜の膜厚 0.27 μm）も同様に、Er:YAG レーザ光の波長 2.94 μm で干渉波形の谷の部分になり、Er:YAG レーザ光を低損失に伝送できると思われる。

D．結論

従来の導光効率を犠牲にした Er:YAG レーザ用の短尺な充実型ガラスファイバに対して、高エネルギー伝送ならびに滅菌工程に耐える超細径中空ファイバが実現できれば、内視鏡治療において、Er:YAG レーザ光を効率よく用いることができる。また導光効率の飛躍的な向上により、レーザ光源の低出力化に繋がり経済性のメリットも生じる。現在、用いられている医療用レーザ装置の短尺な充実型ガラスファイバは、加工工程が複雑なため高価である。一方中空ファイバは簡単な構造で安価に製造でき、消耗品のコストを下げることができるため、医療費の抑制に繋がる。最小侵襲治療が叫ばれている医療現場において、レーザによる低侵襲治療は社会的な要求であり、それに関連する治療装置の開発は極めてニーズが大きい。経済性を考慮しつつ、医療現場において感染症を防止することも重要な課題である。これらの要求を一度に満たす手段として、耐久性の高い無機材料を用い、製作が容易でしかも滅菌可能な、もしくはディスポーザブルな医療用ファイバを実現することは、極めて大きな意味を持つ。また、治療・療養期間の短縮化、高齢者保護の上で、その社会的な効果も十分ある。平成 21 年度に超細径無機ガラス薄膜内装銀中空ファイバの製作を行い、

目標値：内径 0.1 mm、長さ 10 cm、Er:YAG 透過率 60 %

達成値：内径 0.1 mm、長さ 10 cm、透過率 80 %（波長 1.7 μm）

と目標値を達成することができた。無機溶液に OC300 を用い、送液コーティング法について検討を行った。濃度が濃い溶液を安定に送液するために、双方向から溶液に力を加える手法を用い、また接続点における溶液の送液速度の変動を抑制するために、送液装置の改良を行った。濃度 38 wt% の無機溶液を用いることで、Er:YAG レーザ光の伝送に適した無機ガラス薄膜を成膜することができた。本研究で超細径無機ガラス薄膜内装銀中空ファイバが製作できたことにより、歯科内視鏡用内径 0.1 mm 無機ガラス薄膜内装銀中空ファイバの製作が期待できる。

平成 22 年度は、内視鏡に導入するために、超細径無機ガラス薄膜内装銀中空ファイバ（内径 0.1 mm、長さ 10 cm）の先端封止技術の開発を行い、先端封止部の Er:YAG の透過率 70 %を目標とする。また製作した超細径無機ガラス薄膜内装銀中空ファイバの評価を行う。

E．健康危険情報

特になし

F．研究発表

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G. 知的財産権の出願・登録状況
1. 特許取得
特になし
2. 実用新案登録
特になし
3. その他
特になし

H. その他
特になし

研究成果の刊行に関する一覧表

雑誌

発表者氏名	論文タイトル名	発表誌名	巻号	ページ	出版年
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K. Iwai, M. Miyagi, Y. W. Shi, X. S. Zhu, and Y. Matsuura	Fabrication of hollow optical fiber with a vitreous film for CO_2 laser light delivery	Proc. SPIE	Vol. 7173	71730Q-1-71730Q-7	2009

国内学会予稿集

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岩井 克全, 高久 裕之, 宮城 光信, 石山 純一, 本郷 晃史, 石 芸尉	充填コーティング法を用いたAgI内装銀クラッドSUS管先端チップの製作	平成21年度電気関係学会東北支部連合大会		117	2009
岩井 克全, 高久 裕之, 宮城 光信, 石山 純一, 本郷 晃史, 石 芸尉	銀クラッドステンレス管を用いたAgI/Ag中空ファイバの伝送特性	電子情報通信学会通信ソサイエティ大会講演論文集		290	2009

Fabrication and characterization of infrared hollow fiber with multi- SiO_2 and AgI inner-coating layers

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We report the transmission characteristics of infrared hollow fiber with multi- AgI and SiO_2 inner-coating layers in the mid-infrared region. A three-dielectric-layer hollow glass fiber with a SiO_2–AgI–SiO_2–Ag structure was fabricated and low-loss property was obtained in the mid-infrared region. The SiO_2 films were coated by use of the liquid-phase coating method and a semi-inorganic polymer was used as the coating material. For deposition of the AgI film between the two SiO_2 films, a silver film was first plated by use of the silver mirror reaction method. Then the iodination process was conducted to turn the silver layer into silver iodide. A calculation method was also developed to estimate the film thickness of dielectric layers in each fabrication step according to the position of loss peaks in the measured loss spectra. Good agreement between calculated and measured loss spectra was demonstrated by taking into consideration material dispersion and surface roughness of inner-coating films.

OCIS codes: 060.2390, 230.4170, 040.6040, 160.5470, 260.2030.

1. Introduction

Hollow fiber [1–3] with inner coatings of a silver layer and a dielectric layer obtains low-loss property in infrared regions. It is capable of delivering key infrared lasers that are widely used in medicine and industry, including Nd:YAG, Ho:YAG, Er:YAG, CO, and CO_2 lasers. The low-loss property can be attained at a target wavelength by selecting an appropriate dielectric film thickness according to the design theory [4]. Furthermore, to increase the number of dielectric layers is also an effective means to lower the attenuation of the hollow fiber.

The structure composed of periodically deposited dielectric layers of low and high refractive indices [5,6] is one of the most commonly used structures for multiple dielectric layer hollow fibers. However, one encounters many difficulties in fabricating multilayered hollow fiber. One is the fabrication technique for a uniform and durable hollow fiber. For the application of a conventional glass-drawing technique, it is not easy to find two appropriate optical materials that can maintain the original structure after the co-draw process. Other fabrication methods that involve sputtering, electroplate coating, chemical vapor deposition (CVD), and liquid-phase chemistry are proposed. Recently, several different materials were used for multilayer hollow fiber fabrication. Both PbS–CdS [7,8] and Ta_2O_5–SiO_2[9] pairs were proposed, and fabricated three-layer hollow fiber showed low-loss property in the near-infrared regions. Limited by the coating techniques, thick films tend to have a rough surface and high nonuniformity. To avoid additional loss caused by the imperfections, these multilayer fibers need thin films targeted at near-infrared regions for Nd:YAG lasers.

We report the fabrication of multilayer hollow fiber with a SiO_2–AgI–SiO_2–Ag structure. AgI and SiO_2 were chosen because they have been studied

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extensively with regard to hollow fibers and the coating techniques have been well established. We carried out research using these two materials separately [10,11]. Coating techniques have been improved to obtain smooth and uniform optical films in the hollow air core surface. Furthermore, it is possible for the coating process to avoid high temperature curing that could destroy the flexibility of a hollow glass fiber. The calculated loss spectrum of multilayer hollow fiber was used to estimate the film thickness of each layer. Loss spectrum in each fabrication step for the SiO_2–AgI–SiO_2–Ag hollow fiber was measured and good agreement with the calculated results was demonstrated when we took into consideration material dispersion and surface roughness of inner-coating films.

2. Theoretical Loss Spectrum

The theoretical calculation for loss spectrum is based on the ray-optics theory [12]. For a hollow fiber of length z, the transmitted power $P(z)$ is

$$P(z) = \int_0^{\theta_{\max}} P_0(\theta) \exp\left[-\frac{1-R(\theta)}{2T\cot\theta} z\right] \sin\theta d\theta, \quad (1)$$

where $P_0(\theta)$ is the angular distribution of the incident beam, $R(\theta)$ is the power reflection coefficient, T is the inner radius of the hollow core, and $\theta_{\max}$ denotes the maximum launching angle. For multilayer hollow fiber, $R(\theta)$ can be calculated from characteristic matrix M:

$$M = \begin{bmatrix} m_{11} & m_{12} \\ m_{21} & m_{22} \end{bmatrix} = \begin{bmatrix} \cos(k_0 nz \cos\theta) & -\frac{i}{p}\sin(k_0 nz \cos\theta) \\ -ip \sin(k_0 nz \cos\theta) & \cos(k_0 nz \cos\theta) \end{bmatrix}, \quad (2)$$

where $p = \sqrt{\frac{\varepsilon}{\mu}}\cos\theta$ for the TE mode and $p = \sqrt{\frac{\mu}{\varepsilon}}\cos\theta$ for the TM mode [13]. Then the reflective coefficient can be expressed as

$$r = \frac{(m_{11} + m_{12})p_l - (m_{21} + m_{22}p_l)}{(m_{11} + m_{12})p_l + (m_{21} + m_{22}p_l)}. \quad (3)$$

With regard to the multiple-layer calculation, matrix M_{total} can be calculated as

$$M_{\text{total}} = M_1(z_1)M_2(z_2 - z_1)M_n(z_N - z_{N-1}), \quad (4)$$

where M_1 is the characteristic matrix of the dielectric layer closest to the air core of hollow fibers, M_2 is that of the layer beneath the former layer, and M_n is that of the layer on the metal layer. Then the equivalent $R(\theta)_{\text{total}}$ for multiple dielectric layers can be derived from the equivalent characteristic matrix M_{total}. For the calculation, $R(\theta)$ depends on the refractive index of the dielectric film, and material dispersion has a significant influence on the shape and positions of low-loss valleys of the loss spectra. Therefore, material dispersion [14] for AgI and SiO_2 is also taken into consideration for the loss spectrum calculation.

A. Loss Spectrum Calculation

Figure 1 shows the calculated results for the loss spectra of hollow fiber with a single SiO_2 layer and fifteen dielectric layers (eight SiO_2 layers and seven AgI layers alternately). Both fibers attain low-loss property at the target wavelength of 2.94 μm. The bandgap effect of the multilayer coating can be observed and thus has a much lower loss than that of the single-layer coating hollow fiber. The calculated results also show that loss at the target wavelength decreases dramatically with an increase in the number of dielectric layers. When the number is greater than 100, loss does not decrease quickly, because the bandgap effect has been well established by the high reflection rate of the outer metallic layer [9].

B. Film Thickness Estimation

The optimized film thickness is important to realize low loss at a target wavelength. For single-layer hollow fiber, the film thickness can be easily calculated according to the wavelength of interference peaks in the measured loss spectrum [4]. For multilayer hollow fiber, it is impossible to simply calculate the film thickness of each layer using a certain formula. We used the above-mentioned loss spectrum calculation method to estimate the thickness of each layer. When calculated and measured loss spectra are in good agreement, we conclude that the actual film thickness is equal to the thickness value in the calculation. Fabrication parameters that control the film thickness should be carefully adjusted according to the estimated results so that optimized film thicknesses can be obtained. As we have shown in previous publications [11,14], the shape of the loss spectra relies greatly on the dispersion property of film material. For the calculation, we used the following Cauchy formula for SiO_2 and AgI material derived in [14]:

$$\text{AgI}: n(\lambda) = 2.0216 + 0.0878/\lambda^2 - 0.0024/\lambda^4, \quad (5)$$

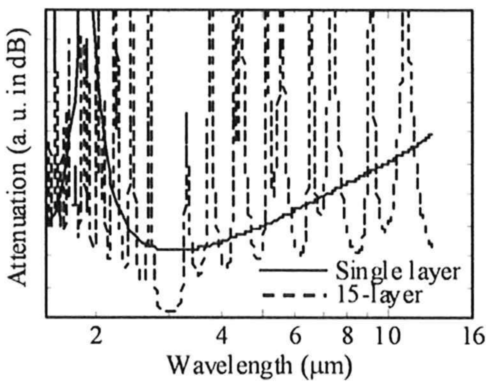

Fig. 1. Calculated loss spectra of hollow fiber with single and fifteen dielectric films.

$$SiO_2 : n(\lambda) = 1.42614 + 0.02729/\lambda^2 + 0.0001/\lambda^4. \tag{6}$$

Figure 2 shows the calculated loss spectra of hollow fiber with a SiO_2–AgI–SiO_2–Ag structure for Er: YAG laser delivery at 2.94 μm wavelength. The loss spectrum is shown when the three dielectric layers are all with the optimized thicknesses. When there is an error for the inner-coating layer thickness, the loss spectrum changes. Figure 2 also shows the calculated results when one of the three inner-coating layers becomes 20% thicker than the optimum. When the first layer (SiO_2) on the silver layer is 20% thicker, the loss spectrum is shown as the first layer in the figure. When the 2nd layer (AgI) or the third layer (SiO_2) is thicker, the loss spectra are shown as the 2nd layer and the 3rd layer in the figure, respectively. Considering the loss property at the target wavelength of 2.94 μm, we note that the second layer (the middle AgI layer) has the strongest impact on the transmission property when it has a thickness error. The first SiO_2 layer on the Ag film has the largest tolerance on the thickness error.

C. Surface Roughness

Limited by the film coating techniques, surface roughness exists for each layer. Normally, a thicker film has a rougher surface. The roughness causes additional loss whose property depends on the wavelength. Light reflected by a rough surface can be divided into two parts: diffuse reflection and specular reflection. By considering that the surface RMS roughness σ is much smaller than the operating wavelength, for simplicity, it is assumed that inclination of the surface is minimal and the energy of diffuse reflection could be disregarded. The effect of roughness on the reflected light can be regarded as the phase offset of the specular reflection of light [15]:

$$\Delta\phi = 2k_0 n_i \sigma \cos\varphi_i, \tag{7}$$

where φ_i represents the angle of incidence. Then the reflectance coefficient r on the rough surface can be expressed by the coefficient of the perfectly smooth surface r^0 [15]:

$$r = r^0 \exp[-(\Delta\phi)^2/2]. \tag{8}$$

Use of Eq. (8) for calculation of the theoretical loss spectrum yields the effect of roughness. Figure 3 shows the theoretical loss spectrum (black curve) of SiO_2–AgI–SiO_2–Ag hollow fiber when the three dielectric layers have perfectly smooth inner surfaces. Theoretical loss spectra were also added for comparison when one of the three layers had a rough surface. The green, red, and blue curves correspond to the calculation results when the first, second, and third layers have a RMS surface roughness of 30 nm. Note that, when the surface is rough, not only the loss increases but also the position of low-loss valley shifts to longer wavelengths. Similar to the influence of the film thickness error, the second layer of AgI has the strongest impact on the transmission property. The first SiO_2 layer on the silver film has the weakest influence. Therefore, much attention should be paid to the AgI coating during the fabrication process because the sensitivity of the transmission characteristics is more dependent on the property of the middle AgI film.

3. Experiments and Discussion

A. Fabrication Process

A glass capillary was used as the supporting tube for the hollow fiber because of its smooth inner surface and thus the potential for high quality optical film coatings. We fabricated a three-dielectric-layer hollow fiber with the structure of SiO_2–AgI–SiO_2–Ag. The thicknesses for each dielectric layer from air core to the metal layer are 0.75, 0.72, and 1.02 μm, respectively. Four steps are necessary for the fabrication process: the inner coating processes for Ag–SiO_2–AgI and SiO_2 films in sequence.

First, a Ag layer was deposited on the inner wall of the capillary tube by the liquid-phase chemistry method. The thickness of the silver layer was approximately 100 nm. A thinner layer is preferred

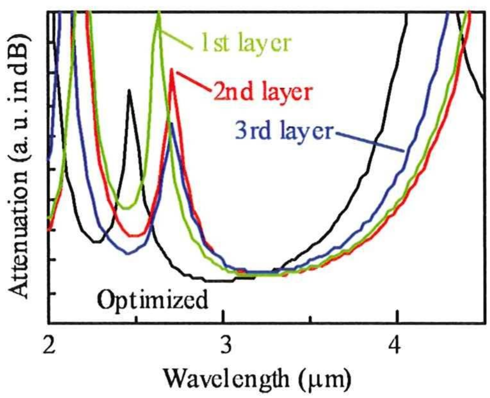

Fig. 2. (Color online) Calculated loss spectra of hollow fiber with and without thickness errors for each inner-coating layer.

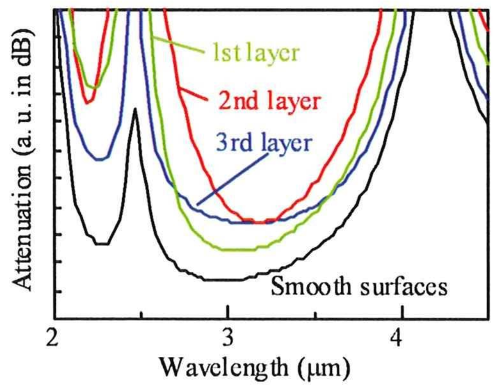

Fig. 3. (Color online) Theoretical loss spectra of Ag–SiO_2–AgI–SiO_2 hollow fiber with and without surface roughness for each inner-coating layer.

because it has less surface roughness. Second, we used OC300 [10] as the coating solution to form a SiO_2 layer on the silver layer. The OC300 solution is a semi-inorganic polymer based on the structural unit R_2SiO. The advantage of this material is that it can be formed at room temperature. Third, deposition of the AgI layer on the SiO_2 film, which can be divided into two separate parts: one part can be used to deposit a silver layer on the SiO_2 layer, the other is used to turn the silver layer into a silver iodide layer by the process of iodination. Finally, a process similar to the second one was used to form another SiO_2 layer on the AgI layer. By comparing the measured and the calculated loss spectra, we estimated the film thickness after each inner-coating process. Fabrication parameters that control the film thickness were carefully adjusted to obtain the expected film thicknesses.

B. Loss Property

The loss spectrum for each fabrication step is shown in Fig. 4. Calculated results based on the model developed in Section 2 are also added. By use of the fabrication process mentioned in Subsection 3.A, the silver and three dielectric layers were coated in order on the inner surface of the glass capillary tube.

Figure 4(a) shows the loss spectrum of the hollow fiber after the Ag and the first SiO_2 layer are deposited. The interference peak at the wavelength of 3 μm shows that the SiO_2 layer is 0.75 μm thick. The loss peak in the 8–10 μm wavelength band is the Si—O bond absorption in the thin film. The sharp peak at the 3.5 μm wavelength is the C—H bond absorption that is caused by the impurity in the silicone polymer. Figure 4(b) shows the hollow fiber with a Ag–SiO_2–Ag structure. The loss spectrum is typical for a Ag tube. Because the Ag layer on the SiO_2 film is thicker than the skin depth of the light and the SiO_2 layer provides minimal contribution to the optical property of the hollow fiber, no Si—O bond absorption can be observed, which means that no light penetrates the silver layer and transmits to the SiO_2 layer and is then reflected back to the air core. There are several sharp pulses in the spectrum that are caused by the noise of the measuring system and have no relation to the loss property. These sharp noise pulses are also shown in Fig 4(d).

The Ag layer near the air core in Fig. 4(b) was turned into a AgI layer during the next fabrication process. Figure 4(c) shows the hollow fiber with the AgI–SiO_2–Ag structure, which is the loss spectrum with two dielectric layers. The typical absorption of the Si—O bond can be observed in the loss spectrum, which means that light penetrates the AgI and SiO_2 layers and is then reflected back to the air core by the bottom Ag layer. The theoretical loss spectrum shows that the AgI layer is 0.72 μm thick. According to our calculations, there is an interference peak at 9.5 μm wavelength, which overlaps with the Si—O bond absorption.

Figure 4(d) shows the loss spectrum of the hollow fiber after the final coating process for the SiO_2 layer near the air core with the structure of SiO_2–AgI–SiO_2–Ag. We observed more high-loss peaks due to thin-film interference on both sides of the low-loss band around 5 μm. In comparison with the calculated results, we note that the Si—O bond absorption

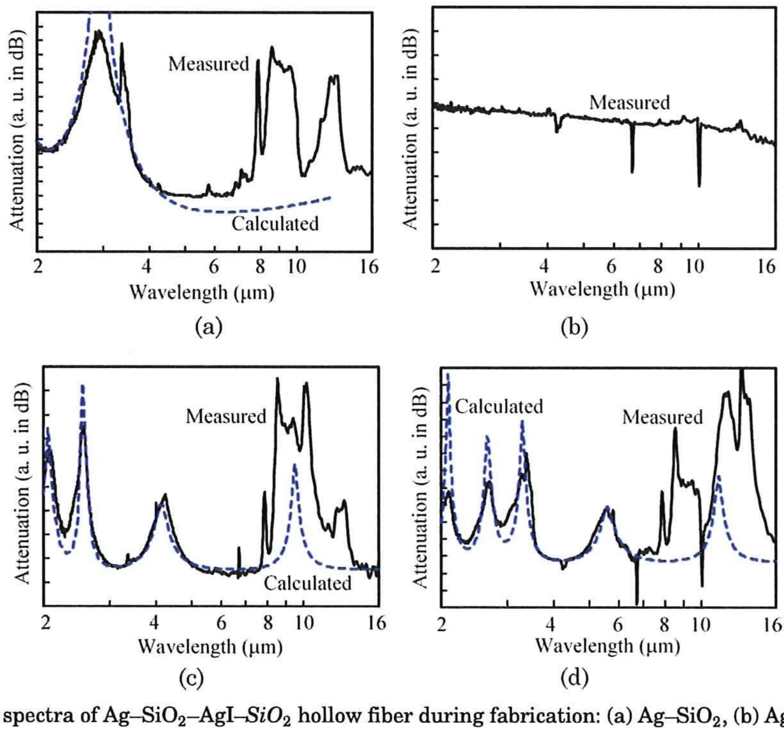

Fig. 4. (Color online) Loss spectra of Ag–SiO_2–AgI–SiO_2 hollow fiber during fabrication: (a) Ag–SiO_2, (b) Ag–SiO_2–Ag, (c) Ag–SiO_2–AgI, and (d) Ag–SiO_2–AgI–SiO_2.

appeared at the loss valley; the shape of typical Si—O bond absorption can be clearly seen.

Theoretically, the loss of the fiber decreases with the increase in dielectric layers. The loss in Fig. 4(d) with three layers is approximately 1 dB smaller than that in Fig. 4(a) with a single layer. The loss differences are not exactly the same as the calculated results in Fig. 1 because each inner-coating layer has a certain surface roughness. Based on the previous fabrication results for hollow fiber with AgI–Ag and SiO_2–Ag film structures, AgI film has a rougher surface than that of a SiO_2 film. However, for the multilayer inner coating, the surface roughness of a film also depends on the surface status of the previously coated layer. The baseline of the loss spectrum moves up and down in each coating process due to the roughness of each coating layer.

4. Conclusions

Transmission characteristics of infrared hollow fiber with multi- AgI and SiO_2 films have been discussed experimentally and theoretically. Three-dielectric-layer hollow fiber with the SiO_2–AgI–SiO_2–Ag structure was fabricated and low-loss property in the mid-infrared regions was obtained. Fabrication parameters were carefully adjusted according to thickness estimation by use of the loss spectrum calculation model. Theoretical calculation results for each fabrication step demonstrated good agreement with the measured data when material dispersion and surface roughness of dielectric film are taken into consideration.

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Fabrication and transmission characteristics of infrared hollow fiber based on silver-clad stainless steel pipes

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Silver-clad stainless steel pipe is used as the supporting tube for the fabrication of infrared hollow fiber. The hollow fiber has high mechanical strength and is highly durable for use in the medical sterilization process. Film of a cyclic olefin polymer layer or silver iodide (AgI) was coated internally to reduce the transmission loss. A liquid-filling method is proposed for coating the AgI layer. Multiple coating processes proved to be effective to increase the AgI film thickness. A treatment of sodium thiosulfate water solution is also proposed to reduce the film thickness. The film thickness can be accurately controlled by combining the coating and decoating techniques. A loss of less than 0.2 dB was obtained for CO_2 laser light for a hollow pipe with a length of 280 mm and an inside diameter of 0.75 mm. © 2009 Optical Society of America

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1. Introduction

CO_2 laser radiation at a wavelength of 10.6 μm has found applications in industrial processing owing to its high output power. It is also one of the most commonly used lasers in the medical field for laser surgery because of its high water absorption coefficient at 10.6 μm wavelength. The laser power transmission system has been critically needed and actively developed [1–6] because of the wide application of the CO_2 laser. We developed hollow fiber with polymer and silver inside-coating films based on a glass capillary with high flexibility. Low-loss infrared hollow fibers with inner diameters from 320 to 1000 μm and a length of 2 m were successfully developed [6].

In medical applications, such as dentistry and otorhinolaryngology, a sterilization process must be done for the recycled output probe of the infrared laser delivery system. It has been shown that the dielectric-coated silver hollow tips were damaged after several sterilization cycles [7] by use of an autoclave. It was observed that the coated silver layer was detached from the inner surface of the glass capillary, which is caused mainly by the rather thin Ag film. To obtain a smooth silver surface for better optical film, the silver film had to be thin [8], which is normally from 50 to 200 nm thick.

Here we use metal supporting pipes for fabrication of the infrared hollow fiber [9]. Fabrication and

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transmission characteristics of infrared hollow fiber based on stainless steel pipes are experimentally discussed. Cyclic olefin polymer (COP) [10] and silver iodide (AgI) [11] were used as the dielectric coating material. COP is a nontoxic, transparent material in the infrared region and can be used to form high quality optical film. AgI is a traditional infrared material that is suitable for high power delivery because of its good adhesion with silver film and its heat resistance. These two kinds of dielectric film were used in the hollow output probe in the fabrication; the transmission properties were experimentally evaluated.

2. Supporting Tube

We used a silver-coated glass capillary as the metallic hollow fiber because of its smooth inner surface. To increase durability for the sterilization process, we propose to use a metal hollow fiber. There are several kinds of supporting tube that have the potential for high durability and are commercially available. They are stainless steel pipe (SUS) with a smooth polished inner surface, gold-coated stainless steel pipe (Au/SUS), and silver-clad stainless steel pipe (Ag/SUS). (SUS is the code name assigned to stainless steel by the Japanese Industrial Standard.) The Ag/SUS tube is made by inserting a silver tube into a stainless steel tube or by using the extrusion process to simultaneously form the silver and the stainless dual-clad pipe. The inner wall of the Ag-clad tube was carefully polished. The silver tube has a wall thickness of 100 μm and is expected to be of high durability and low loss when inner coated with dielectric optical films. The Au-coated tube is fabricated by electroplating. The longest length made up to now is 280 mm. The length for a Ag-clad tube is limited to approximately 1 m because of the polishing techniques. Both Ag-clad and Au-coated tubes are custom-made products.

Figure 1 shows the structure of various metal pipes. Table 1 summarizes the size and transmission loss for CO_2 laser light of the metal pipes. Figure 2 shows the loss spectra for the metal pipes from the visible to the mid-infrared regions. The loss spectrum for a silver-coated glass capillary (Ag/SiO_2) was also added for comparison. Loss spectra for wavelengths less than or greater than 1.5 μm were measured with a spectrum analyzer and Fourier transform infrared spectroscopy (FTIR), respectively. SUS pipe has the highest loss because of its rough inner surface. Au/SUS pipe has a lower loss because of the gold film coating. The silver-clad Ag/SUS pipe

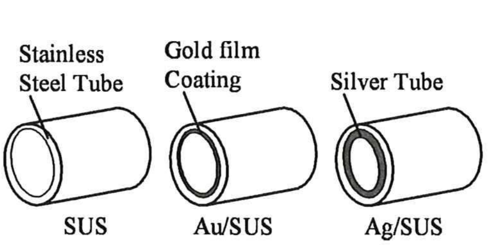

Fig. 1. Structure of various metal pipes.

Table 1. Size and Loss of a CO_2 Laser for Metal Pipes

Material	Size (mm) Inner Diameter	Outer Diameter	Length	Loss (dB)
SUS	0.94	1.2	285	3.7
Au/SUS	0.94	1.2	285	2.1
Au/SUS	0.75	1.2	280	1.7
Ag/SiO_2	0.75	0.9	280	1.8

in the SUS tube has the lowest loss. The property of the Ag/SUS pipe is the same as that of the silver-coated glass tube (Ag/SiO_2) in the mid-infrared region. The measured results are also shown in Table 1. Loss for CO_2 laser light is 1.7–1.8 dB for the Ag/SiO_2 and Ag/SUS pipes, respectively. During the measurement for CO_2 laser light delivery, a coupler was used to minimize the coupling loss between the laser light and the tube. The coupler was a Ag-coated hollow glass fiber with the same inner diameter as the measured tube. However, Ag/SiO_2 has low loss in the visible and near-infrared regions because the glass capillary has a smooth inner surface that guarantees a smooth Ag-layer plating and thus causes smaller additional loss in the shorter wavelength region. During fabrication for the metal pipes, a polishing process is needed to smooth the inner surface for optical film coating. According to observation with an atomic force microscope (AFM), the measured inner surface roughness has a 2 nm root mean square (rms) for a glass capillary and 50 nm for a well-polished stainless steel tube. Therefore, fabrication for long pipes is difficult. A short pipe has strong mechanical strength and is capable of withstanding sharp bending. A short pipe is adequate and safe for application as an output probe for the infrared delivery system.

3. Polymer Inner Coating

Optical film coating on the metal surface can dramatically increase the reflectance rate of the surface. Therefore, it is commonly used to coat a dielectric film on the inner surface of a metal pipe to reduce transmission loss. COP and AgI are two successful dielectric coating materials for infrared hollow fiber.

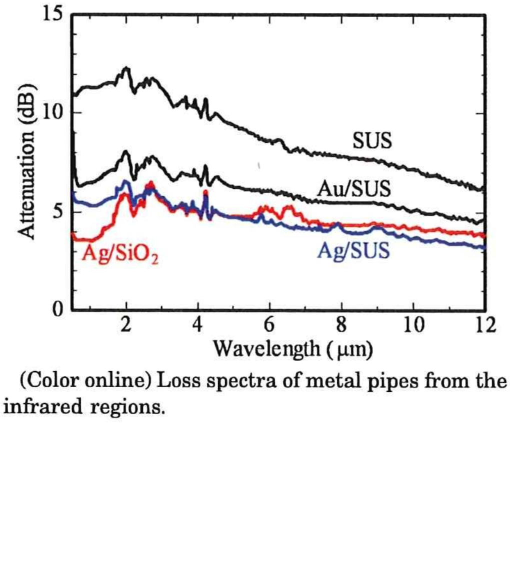

Fig. 2. (Color online) Loss spectra of metal pipes from the visible to the infrared regions.

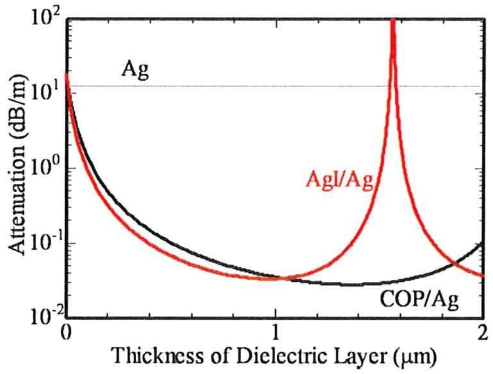

Fig. 3. (Color online) Theoretical losses of the HE_{11} mode in hollow fiber as a function of dielectric film thickness at a wavelength of 10.6 μm.

For a target wavelength, there is an optimum film thickness [12] for each dielectric material.

Figure 3 shows the theoretical loss of the HE_{11} mode in the dielectric-coated hollow fiber as a function of dielectric film thickness. The target wavelength is 10.6 μm for CO_2 laser light. For the calculation, the complex refractive index for Ag at the wavelength of 10.6 μm is $13.5 - j75.3$. The refractive indices for COP and AgI are 1.53 and 1.95 [13], respectively. We note in Fig. 3 that the optimum thicknesses for a minimum loss are 1.36 and 0.98 μm for COP and AgI layers, respectively. The loss of COP/Ag hollow fiber is slightly less than that of the AgI/Ag hollow fiber because the optimum refractive index for the dielectric layer is theoretically 1.41.

We used the COP, dissoved in cyclohexane, as the dielectric coating material. The solution was forced to flow through the metal pipe and a liquid-phase film was formed on the inner surface. After the curing process, the film was solidified to form a stable polymer layer. Detailed fabrication techniques and parameters to control film thickness have been published elsewhere [14]. Figure 4 shows the loss spectra for COP-coated metal pipes. The COP film thickness had been optimized for 10.6 μm wavelength. Table 2 summarizes the loss for CO_2 laser light of the COP-coated metal pipes.

In Fig. 4, loss peaks in the less than 5 μm wavelength region came from the interference effect of

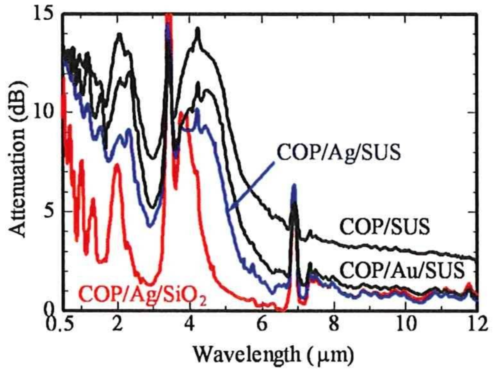

Fig. 4. (Color online) Loss spectra of COP-coated metal pipes.

Table 2. Loss of COP-Coated Metal Pipes for a CO_2 Laser

Material	Size (mm) Inner Diameter	Outer Diameter	Length	Loss (dB)
COP/SUS	0.94	1.2	285	0.6
COP/Au/SUS	0.94	1.2	285	0.22
COP/Au/SUS	0.75	1.2	280	0.18
COP/Ag/SiO_2	0.75	0.9	280	0.13

light in the thin COP optical film. According to the position of the interference peaks, the film thicknesses for the COP layer can be calculated. For the four kinds of COP-coated metal pipe, the film thickness ranges from 0.85 to 0.95 μm. In comparison with the theoretical optimum film, the coated film thickness is thinner. Because thicker film has a much rougher surface that causes additional loss, a thinner film is normally preferred for the fabrication process. Table 2 shows the measured results for CO_2 laser light. A low loss of around 0.2 dB was obtained for the hollow pipes, except for the COP/SUS pipe because the SUS pipe has a rough surface, which had been observed in the loss spectrum in Fig. 2.

4. Silver Iodide Coating

A. Liquid-Filling Method

The coating technique for the AgI layer is an iodination process that changes part of the Ag layer into a AgI layer. The iodine cyclohexane solution flows through the Ag-coated tube and the upper surface of the Ag layer changes into AgI. This AgI coating technique has worked well for hollow fiber with inner diameters ranging from 0.25 to 0.7 mm [15]. However, a rather high flow rate is necessary in the iodination process. To form the AgI layer, a 20 ml iodine solution is needed. We propose a new method that significantly reduces the waste of the iodine solution.

Figure 5 shows the new fabrication method. A syringe is used to fill the Ag/SUS tube with the iodine solution. The tube is completely filled, the solution is kept for a period of time for AgI layer formation, and then the iodine solution is flushed out. By using the newly developed method, the volume of the iodine solution is only 0.1 ml for the 280 mm long metal pipe with a 0.75 mm bore diameter. During the fabrication process, the iodine solution was made by dissolving iodine pellets in cyclohexane. Then the solution was stirred in a supersonic device for 10 min to mix the solution.

Figure 6 shows the AgI film thickness versus liquid-filling time. The concentrations of the iodine solution are also shown. The dashed line indicates the optimum film thickness for the Er:YAG laser that irradiates at 2.96 μm. The fabrication parameter for Er:YAG hollow pipe could have a 60 s filling time for an iodine solution with 0.5% concentration. The film thickness tends to be thicker when the filling time is prolonged. However, a saturation time exists for a certain concentration as can be observed in Fig. 6