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D型高双折射光子晶体光纤的折射率传感特性研究

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摘要:基于表面等离子体共振(SPR)原理,分析了D型高双折射光子晶体光纤(HB-PCF)的折射率传感特性。模拟研究了抛磨角度对双折射、折射率传感灵敏度的影响。仿真结果表明:当抛磨面离纤芯的高度小于1.5倍占空比时,抛磨面离纤芯越近,双折射越小;随着抛磨角度的增加,双折射先增加后减小,折射率传感灵敏度随之减小;当抛磨角度为 0° 且折射率范围在1.330~1.390时,D型HB-PCF的平均折射率灵敏度高达2 833.33 nm/RIU。此外,制备了一种D型高双折射率光子晶体光纤SPR传感器,实验测得其折射率灵敏度约为1 711.83 nm/RIU。D型HB-PCF SPR传感器可应用于生物、化学和环境监测等领域。

关键词:光子晶体光纤;表面等离子体共振;折射率;传感器;侧边抛磨

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0 引言

表面等离子共振(Surface Plasmon Resonance, SPR)是一种物理现象,当入射光的频率与波数与金属表面振动的自由电子(即等离子)频率一致时,则金属表面的电子(即等离子)吸收光能发生共振,其共振波长随贵金属表面折射率变化而变化,而折射率的变化又与贵金属表面结合的分子质量密切相关^[1-7]。因此SPR在生命科学、医疗检测、药物筛选、环境监测、食品检测等领域具有广泛的应用需求^[8-11]。1902年,美国著名物理学家WOOD R W^[12]首次参观到了SPR现象,1941年^[13],科学家解释了SPR现象的物理原理。1968年,KRETSCHMANN E^[14]提出了两种激发SPR的棱形结构,为SPR传感技术的应用奠定了实验基础。1983年,KULLMAN E^[15]将SPR用于IgG与其抗原的反应测定并取得了成功。到了1993年,JORGENSON R等^[16]提出了SPR光纤传感器,为SPR技术更加广泛的应用开启了新的乐章。在过去的二十年中,为提高光纤SPR传感器的性能,人们致力于调整和优化基于光纤SPR传感器的设计。为了提高灵敏度和降低谐振峰的窄半峰全宽,人们用单模光纤替代多模光纤作为传感元件。随后出现的光纤SPR传感器结构,为了允许沉积薄金属(主要是Au或Ag)层,光纤的包层被移除(传感区域),该层支持SPR的激发及其与光纤的光波导模式的相互作用。这些结构包括修饰光纤端面^[17]、锥形光纤^[18]、D形光纤^[19]、光纤光栅^[20]和光子晶体光纤^[21]。

如今,基于微结构光纤的SPR传感器因其在生化传感领域具有广泛的应用前景而越来越受欢迎。然

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而,许多研究人员遇到的困难是在设计微结构光纤 SPR 传感器时很难做到实现等离子体共振模式与纤芯模式的完全匹配及空气孔内分析液难以清理。作为创新的解决方案,LUAN Nannan 等^[22]提出了一种基于微结构光纤 SPR 传感器,该结构以两种特殊设计为特色。特色一是结构设计为 D 形空心光纤,可以降低类高斯纤芯模式的折射率,以匹配等离子模式的折射率。另一个新颖之处在于沉积分析物的方法,即将分析物直接沉积在 D 形平面上,而不是填充纤芯。通过这种方法,获得了令人满意的光谱灵敏度(2 900 nm/RIU)。此后,基于镀膜 D 型光子晶体光纤,CHEN Hailiang 等^[23]设计了一种超宽带宽偏振滤波器,该结构的一个好处是它有助于在光纤波导模式和表面等离子体激元(Surface Plasmon Polaritons, SPPs)模式之间产生多重共振。类似的,TAN Zhixin 等^[24]对深度抛磨的全固态光子晶体光纤 SPR 传感器进行了数值研究,利用相位调制,采用相位询问法,计算得到灵敏度为 9.09×10^4 /RIU,理论值高于常规的波长检测灵敏度。值得注意的是,XIE Qingli 等^[25]实验研究了侧边抛磨深度和传感层厚度对 D 形光子晶体光纤表面等离子体共振传感器性能的影响,发现传感灵敏度随着传感层厚度的增加而增加。但当侧抛深度达到一定范围时,传感器灵敏度略有下降,实验测得在 1.40~1.42 的折射率范围内,传感器最高灵敏度可达 7 381.0 nm/RIU。事实上,对于光纤 SPR 传感器,测量的分析液折射率越高,灵敏度越大。此外,TONG Kai 等^[26]提出了一种基于 TiO₂ 薄膜增强的 PCF-SPR 的生物传感器,折射率灵敏度可达 4 200 nm/RIU。CHEN Qianghua 等^[27]提出了一种基于棱镜结构的具有四层介质的光纤表面等离子体共振的传感器,用于检测溶液折射率变化。GANDHI M A 等^[28]以 D 型 PCF 为基质,设计了三种金属沉积方案,实现了高灵敏度传感,可用于生物、化学检测。

目前基于光子晶体光纤 SPR 传感器的报道,主要是以理论模型的建立和数值仿真为主,实验制备基于光子晶体光纤 SPR 传感器较为困难。本文提出了一种 D 型高双折射光子晶体光纤(High Birefringence Photonic Crystal Fiber, HB-PCF)传感器。理论上,采用有限元(Finite Element Method, FEM)数值分析,仿真分析了抛磨深度、抛磨角度对双折射的影响,研究了折射率传感灵敏度与抛磨角度的关系。实验上,制备了 D 型 PCF-SPR 传感器样品,分别实现了对不同折射率匹配液和不同浓度葡萄糖溶液的检测。

1 结构模型与仿真分析

基于 D 型高双折射光子晶体光纤的 SPR 传感器模型结构示意图如图 1 所示。根据实验采用的高双折射光子晶体光纤,仿真模型的光子晶体光纤由五层空气孔排列组成,包层中第一层含有两个较大的空气孔,其中晶格间距 Λ 为 4.4 μm ,小空气孔直径 d_1 为 2.2 μm ,大空气孔直径 d_2 为 4.5 μm ,抛磨深度为 h ,即光子晶体光纤纤芯到抛磨表面的距离,高双折射光子晶体光纤慢轴与抛磨面之间的夹角定义为抛磨方向 θ 。金薄膜涂覆在光纤平坦的抛磨面上,以便于和待测物体接触。根据以往的理论 and 实验研究,把金膜的厚度(t)设置为 45 nm。仿真中用到的光子晶体光纤背景材料折射率和金的折射率由线性插值的实验数据给出。为了得到侧边抛磨高双折射光子晶体光纤的波导模式,采用有限元法商用软件 COMSOL Multiphysics 并设置完美匹配层边界条件来进行仿真,分析物的折射率设置为 1.330 到 1.400 范围内。波长灵敏度定义为 $S_\lambda = \Delta\lambda_{\text{res}}/\Delta n_a$,其中 λ_{res} 是 D 型光子晶体光纤传感器的等离子共振波长, n_a 是分析物折射率。在波导光学领域,限制损耗由等式(1)计算得出^[29]。

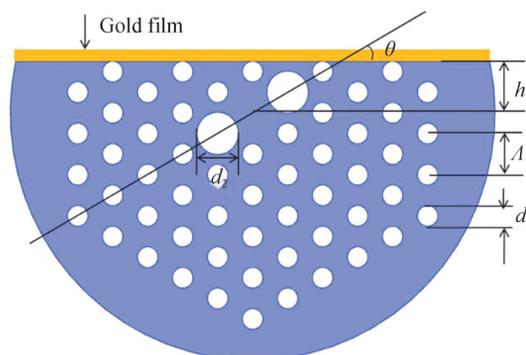


图 1 基于 D 型高双折射光子晶体光纤的 SPR 传感器模型结构示意图

Fig. 1 Schematic diagram of the SPR sensor based on D-shaped high birefringence photonic crystal fiber

$$\alpha = 8.686 \cdot k_0 \operatorname{Im}[n_{\text{eff}}] \text{ (dB/m)} \quad (1)$$

式中, $k_0 = 2\pi/\lambda$ 为传播常数, 单位为 m^{-1} 。

首先研究抛磨对PCF双折射效应的影响。抛磨深度对双折射的影响如图2(a)所示, 抛磨方向设置为 0° , 光波长分别设置为500 nm、1 000 nm、1 500 nm, 由图2可知, 当抛磨面离纤芯的距离大于1.5倍空占比时, 侧面抛磨光纤几乎不对双折射产生影响, 在1~1.5倍空占比之间, 影响较小, 当抛磨面离纤芯距离小于1倍空占比时, 抛磨面离纤芯越近, 双折射越小, 同一抛磨深度, 波长越大, 双折射也越大。图2(b)是抛磨角度对双折射的影响, 很显然, 抛磨方向对双折射的影响较小, 在波长较小时, 其影响可以负略不计。

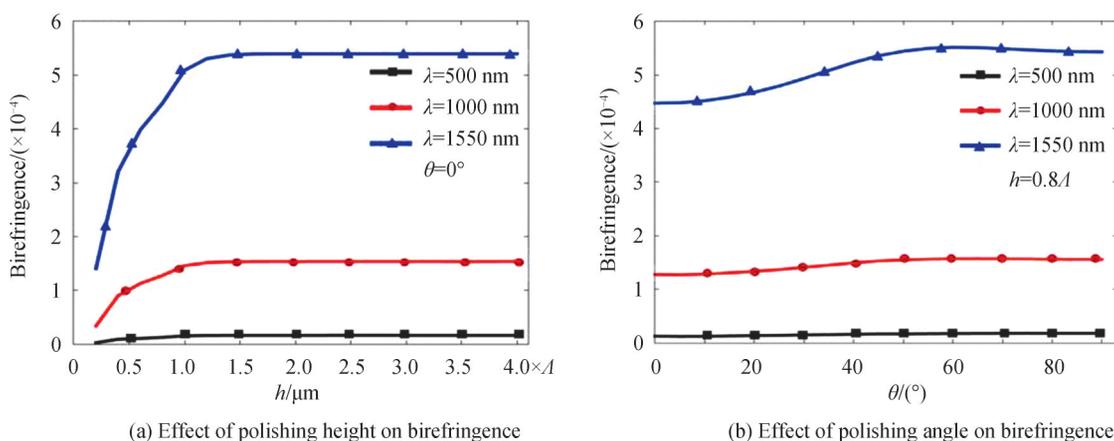


图2 抛磨高度和抛磨角度对光子晶体光纤双折射的影响

Fig. 2 The influence of polishing height and polishing angle on the birefringence of photonic crystal fiber

图3(a)给出了纤芯TM波导模有效折射率、SPPs有限折射率及纤芯TM波导模的损耗与波长的关系。其中, 金纳米薄膜表面待测物折射率设置为1.33, 抛磨角度 θ 为 0° , 抛磨深度 h 为 0.8Λ , d_1 为 $4.5 \mu\text{m}$, d_2 为 $2.2 \mu\text{m}$, Λ 为 $4.4 \mu\text{m}$ 。由图3(a)可知, 纤芯模式有效折射率和SPPs有限折射率随着波长的增加而线性减小, 在波长约为595 nm处, 两者发生相交即纤芯TM模式有效折射率和SPPs有限折射率相等, 满足相位匹配条件下, 此时将发生表面等离子体共振效应, 使得纤芯能量大部分转移到金纳米薄膜层附近, 表现为纤芯导模在此波长有一个损耗峰。为进一步说明该物理现象, 比较了波长在500 nm和595 nm处纤芯导模和等离子体模式的电场分布, 图3(b)为波长 λ 在500 nm处的纤芯TE模, 图3(c)为波长 λ 在500 nm处的纤芯TM模, 图

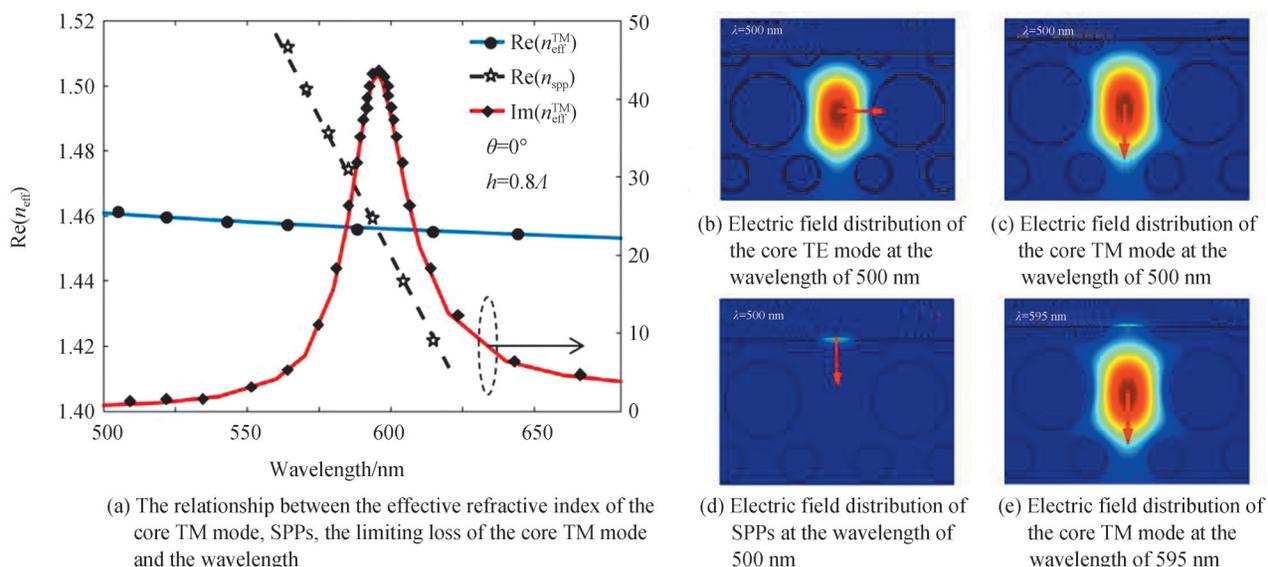


图3 模式的有效折射率及模场分布

Fig. 3 Effective refractive index and mode field distribution of the mode

3(d)为波长 λ 在500 nm处的等离子体模,图3(e)为波长 λ 在595 nm处即达到相位匹配时同时可观察到纤芯导模和等离子体模式。很显然在该波长纤芯 TM 模和等离子体模式发生了显著的耦合。

研究D型高双折射光子晶体光纤的SPR传感特性。其传感原理为:由于SPR对环境折射率变化非常灵敏,当环境折射率变化时,纤芯 TM 模式损耗谱峰值会发生漂移,根据损耗光谱中的峰值波长和待测物折射率之间的关系,利用波长询问法,通过测量损耗光谱的峰值波长来待测物折射率。图4(a)展示了D型高双折射光子晶体光纤的抛磨角度 θ 为 0° 、抛磨深度为 0.2Λ , d_1 为 $4.5\ \mu\text{m}$, d_2 为 $2.2\ \mu\text{m}$, Λ 为 $4.4\ \mu\text{m}$ 时,待测物折射率 n 分别为1.330、1.340、1.350时纤芯 TM 模的损耗谱。随着待测分析物折射率的增加,损耗光谱的峰值波长呈现红移,耦合强度也相应地增加。图4(b)是折射率从1.330变化到1.400时,待测分析物折射率和损耗谱峰值波长、峰值损耗大小的关系,由图4(b)可知,损耗谱的峰值损耗、峰值波长(谐振波长)随着分析液折射率的增加而增大。根据折射率灵敏度的定义,在1.330~1.400折射率范围,计算可得D型HB-PCF的平均折射率灵敏度高达 $3\ 457.14\ \text{nm}/\text{RIU}$,该结果优于D型单模光纤及D型全内反射型光子晶体光纤 SPR 传感器的性能^[29-30]。

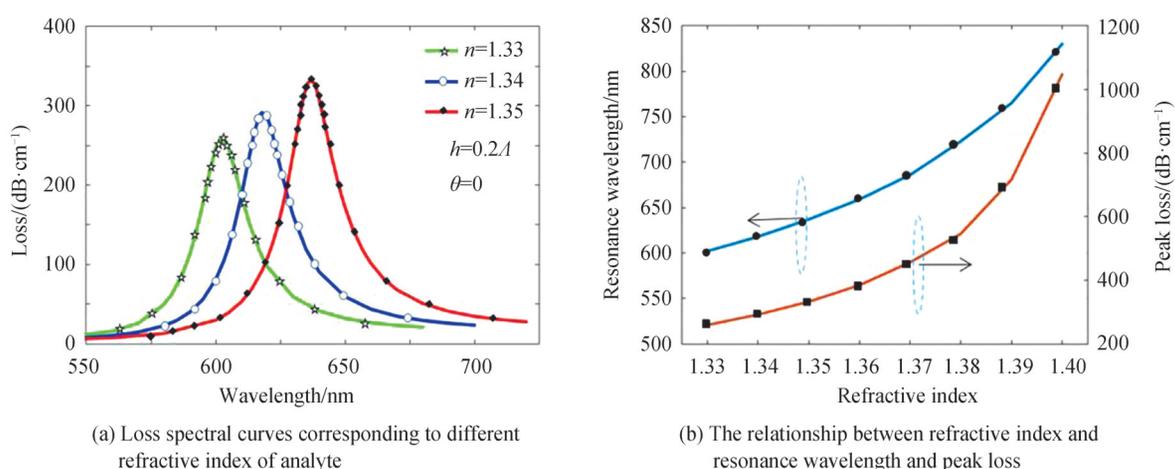


图4 折射率传感理论分析

Fig. 4 Refractive index sensing theory analysis

根据前面的理论和实验研究^[29],光纤的抛磨深度会影响纤芯模式和等离子体模式的耦合强度,但不会影响D型光纤 SPR 传感器的灵敏度,因此只对抛磨角度对D型的HB-PCF SPR 传感器的灵敏度进行分析,如图5(a)所示。图5(a)是抛磨深度为 0.6Λ ,待测分析物折射率 n 分别为1.330和1.400,不同抛磨角度下的D型HB-PCF的损耗谱。由图5(a)可知,其他参数不变情况下,抛磨角度的变化会影响谐振波长的位置。随

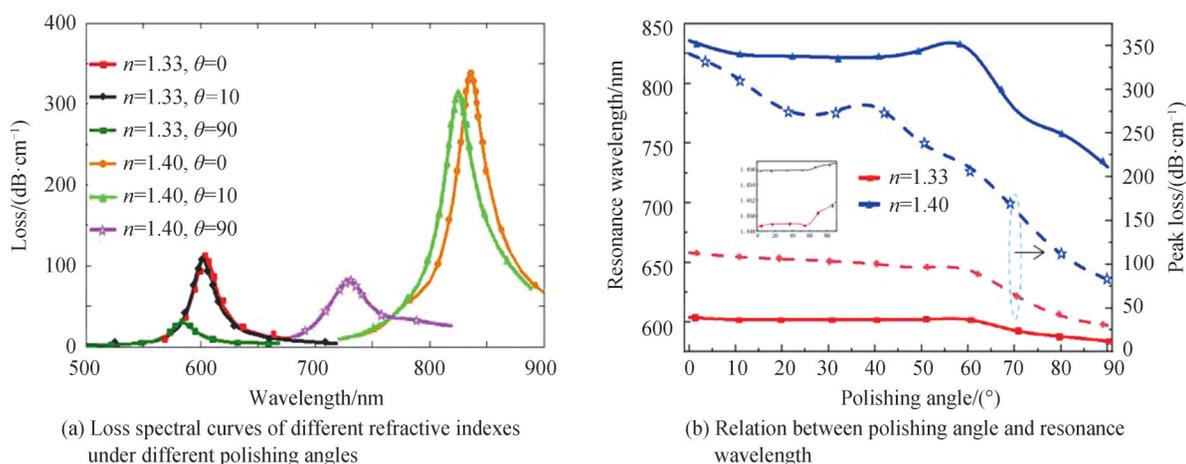


图5 抛磨角度对D型高双折射PCF光传输特性影响

Fig. 5 Effect of polishing angle on the optical transmission characteristics of D-type highly birefringent PCF

着抛磨角度的增大,损耗谱的峰值损耗降低,且损耗峰值波长发生蓝移。图5(b)是谐振波长、损耗谱峰值与抛磨角度的关系。由图5(a)可知,抛磨角度从 0° 逐渐增加到 60° 时,谐振波长漂移变化缓慢,但是在 60° 到 90° 的范围,该过程随着抛磨角度的增加,谐振波长发生显著的漂移,分析液折射率越高,谐振波长受到的影响越大。

2 实验与讨论

实验上,制备了D型高双折射光子晶体光纤 SPR 传感器样品并进行了测试。实验选择由长飞光纤光缆股份有限公司生产的保偏光子晶体光纤(型号:PC1013-A)。图6为光子晶体光侧边抛磨示意图。首先,在两个单模光纤之间熔接一段10 nm长的PCF,然后熔接的光纤由一对光纤夹持器夹住固定。在这个光纤抛磨系统中,砂轮固定在可以沿着 x 、 y 、 z 方向移动的3D机械平台上。抛磨长度和抛磨深度可通过计算机程序精确设置和操作。为了加快抛磨的速度,使用小重量的砝码挂在光纤上来拉直光子晶体光纤并提供合适的抛磨力度。在机械轮上固定砂纸用于抛磨光纤。实时监测抛磨过程用的宽带光源(BBS)波长范围为1 250 nm到1 650 nm,光谱分析仪型号为OSA, Yokogawa AQ6370C。用沾酒精的无尘纸轻轻清洁光子晶体光纤抛磨表面以去除残留的二氧化硅微尘。当抛磨面接近光子晶体光纤的纤芯时,即刻停止抛磨。

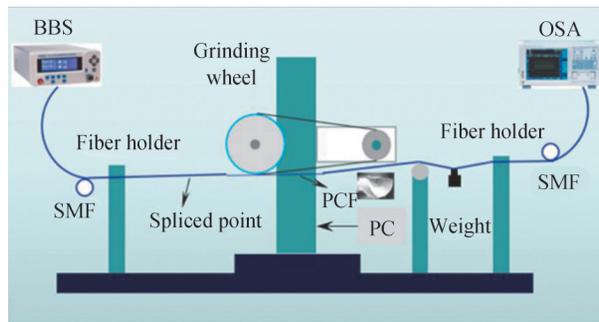


图6 侧边抛磨光子晶体光纤系统示意图
Fig. 6 Photonic crystal fiber side-polished system

在对光子晶体光纤完成抛磨后,通过光纤磁控溅射涂层机(LN-JS2,沈阳真空技术研究所)在抛磨好的光子晶体光纤平坦的表面上沉积45 nm厚度的金薄膜。最后,即可完成侧边抛磨的PCF-SPR传感器的制备工作,抛磨长度为7 mm,抛磨深度为 $0.88 \mu\text{m}$,所获D型高双折射光子晶体光纤在显微镜下观察到的实物如图7所示。

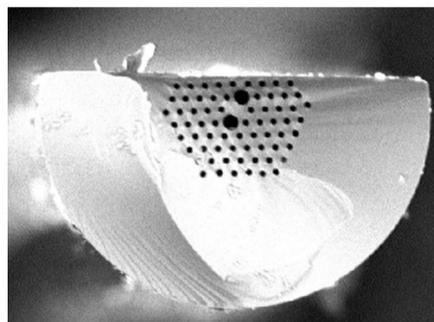


图7 D型高双折射光子晶体光纤横截面示意图
Fig. 7 Cross-section of D-shaped high birefringence photonic crystal fiber

图8为搭建的SPR传感器实验测试装置。在此传感测量系统中,光源采取具有450 nm至1 100 nm宽带的卤钨素灯(LS-300, Ocean Optics Inc)。光谱仪采用一种微型光谱仪(USB6500, Ocean Optics Inc),其测量范围为200 nm至1 100 nm。白光通过单模光纤传输到PCF-SPR传感器中,并受D型平面上折射率匹配液体的调制。最后,在室温下的调制信号由光谱仪记录并通过笔记本电脑的USB端口处理。实验中,需要在

每个后续测量之前用酒精反复清洗传感器以确保感测敏感区域的清洁。

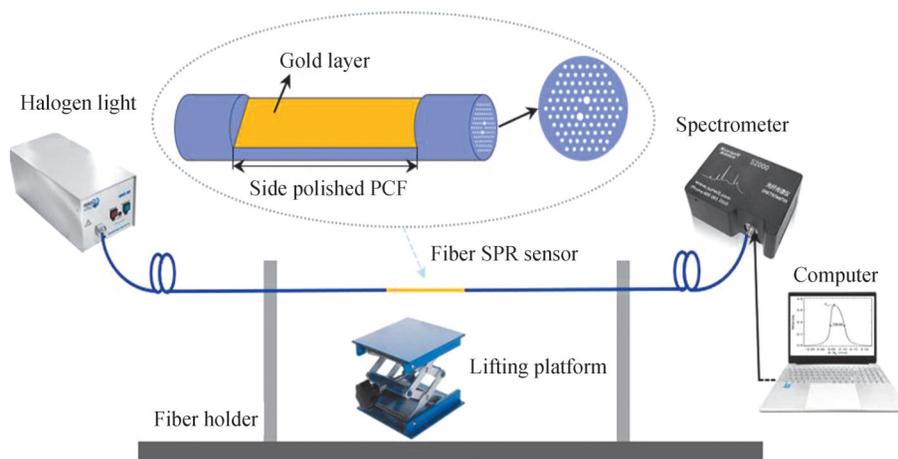


图8 实时在线测量系统的示意图

Fig. 8 Schematic diagram of real-time online measurement system

D型高双折射光子晶体光纤的折射率传感测试结果如图9(a)所示。归一化透射光谱根据传感器浸入液体时的透射率与传感器暴露在空气中的透射率之比来计算。作为电磁场从纤芯TM模耦合到金膜附近谐振波长处SPP模式的结果,当传感器浸入每种折射率匹配液时,传输谱中会出现明显的吸收峰。随着匹配液折射率的增加,传输谱的凹点波长会出现红移,且凹点深度会随着匹配液折射率的增加而增大。图9(b)是谐振波长和分析液折射率变化之间的非线性关系。随着匹配液折射率的增加,D型高双折射光子晶体光纤的折射率灵敏度逐渐增大。计算可得其在1.330~1.390折射率范围的平均折射率灵敏度约为1711.83 nm/RIU。由图4(b)可知,在该折射率范围,其折射率灵敏度理论值为2833.33 nm/RIU。理论和实验上的灵敏度值相差较大,主要原因有:1)抛磨面不平整(空气孔带来的缺陷),使得抛磨过程中产生的碎屑不易完全清除,这会影响到传感器的性能;2)完成D型光纤样品制备后,没有及时对光纤做镀膜处理,D型光纤长时间暴露在空气中,空气中的尘埃附着在D型面会进一步影响器件性能;3)尽管在每次测试之前均会反复用乙醇清洗传感器,但是难以把留在PCF空气孔中折射率匹配液完全清除,从而影响后续测量结果的准确性,例如,第一次测试折射率1.33,通过酒精清洗,滴1.34的光纤匹配液,由于前面的液体残留,真实值很难做到1.34,而计算依旧按照1.34计算;4)理论的最大值是在抛磨平面与两个大空气孔连线平行条件下获得的,理论仿真已表明其灵敏度与抛磨的方向有关,测试样品的抛磨面并不与两个大空气孔的连线平行。

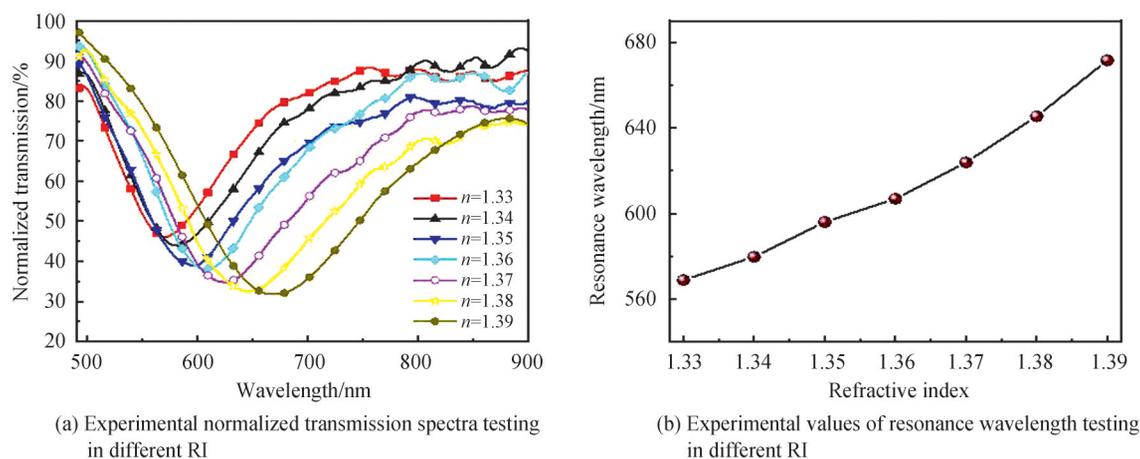


图9 折射率传感实验分析

Fig. 9 Experimental analysis of refractive index sensing

进一步用该D型高双折射光子晶体光纤 SPR 传感器测试葡萄糖溶解的浓度,实验结果如图 10(a)所示。葡萄糖溶液浓度以 2 g/dL 的步长,从 0 g/dL 增加至 10 g/dL,由图 10(a)可知随着葡萄糖浓度的增加,D型高双折射光子晶体光纤 SPR 传感器的传输谱凹点峰值波长会发生红移。在 0g/dL 的葡萄糖溶液中,SPR 共振波长出现在 578.96 nm 处,而当葡萄糖溶液浓度为 10 g/dL 时,SPR 共振波长漂移到 587.49 nm 处。图 10(b)给出了葡萄糖浓度与传输谱凹点峰值波长的关系,经计算,其平均灵敏度为 1.89 nm/(g/dL)。

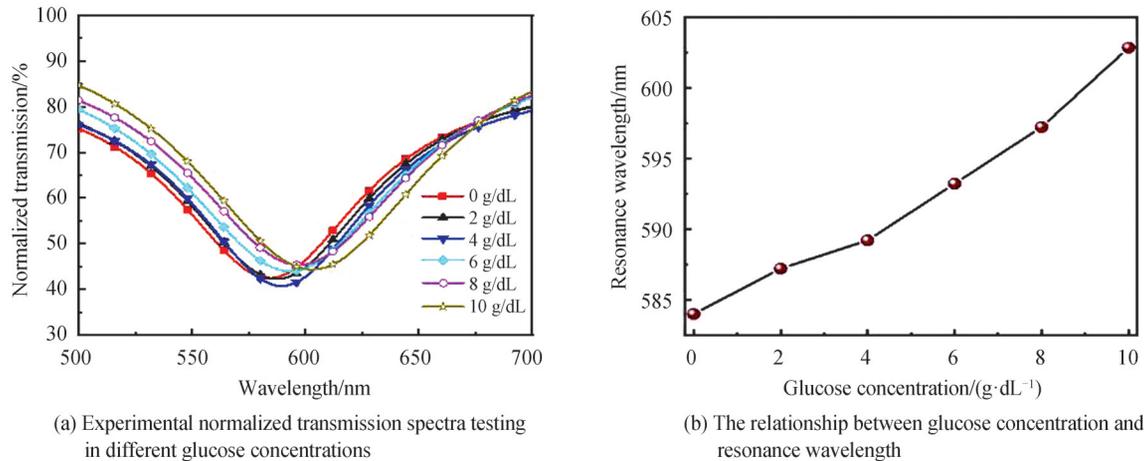


图 10 葡萄糖浓度检测分析
Fig. 10 Glucose concentration detection analysis

3 结论

基于 SPR 原理,本文数值仿真和实验研究了 D 型 HB-PCF 的折射率传感特性。金膜和分析液被沉积在 HB-PCF 抛磨的表面。通过有限元法建模仿真,研究了抛磨角度对双折射、折射率传感灵敏度的影响。仿真结果表明:高双折射光子晶体光纤的双折射效应受抛磨深度的调制;而折射率灵敏度与抛磨角度密切相关;当抛磨角度为 0° 且折射率范围在 1.330~1.400 时,D 型 HB-PCF 的平均折射率灵敏度可达 3 457.14 nm/RIU。实验制备了 D 型 HB-PCF SPR 传感器,并测试了其折射率传感。D 型高双折射光子晶体光纤 SPR 传感器可以实现在生物、化学和环境监测等领域的应用。

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Refractive Index Sensing Characteristics of D-shaped High Birefringent Photonic Crystal Fiber

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Abstract: Surface Plasmon Resonance (SPR) is a physical phenomenon, when the frequency and wave number of incident light coincide with the frequency of free electrons vibrating on the metal surface, then the electrons (i.e., plasma) on the metal surface absorb light energy and resonate, and its resonance wavelength changes with the refractive index of the precious metal surface, so SPR has a wide range of application needs in medical detection, environmental monitoring and other fields. Based on the principle of SPR, this work investigates the refractive index sensing characteristics of D-type highly birefringent photonic crystal fibers in detail. The current reports on PCF SPR sensors are mainly based on the establishment of theoretical models and numerical simulations. It is difficult to experimentally prepare PCF SPR sensors. According to the high birefringence photonic crystal fiber used in the experiment, the photonic crystal fiber of the simulation model is composed of five layers of air holes. The first layer of the cladding contains $2.2\ \mu\text{m}$, the diameter of the large air hole is $4.5\ \mu\text{m}$, and the polishing depth is represented by h , that is, the distance from the core of the photonic crystal fiber to the polishing surface, and the angle between the slow axis of the high birefringence photonic crystal fiber and the polishing surface is defined as the polishing direction θ . The gold film is coated on the flat polishing surface of the optical fiber to facilitate contact with the object to be measured. According to previous theoretical and experimental research, we set the thickness (t) of the gold film to $45\ \text{nm}$. The refractive index of the photonic crystal fiber background material and the refractive index of gold used in the simulation are given by the experimental data of linear interpolation. In order to obtain the waveguide mode of the side-polished high-birefringent photonic crystal fiber, this paper uses the finite element method commercial software COMSOL Multiphysics and sets the boundary conditions of the perfect matching layer for simulation. The refractive index of the analyte is set in the range of 1.330 to 1.400 . Through finite element method modeling and simulation, the influence of polishing angle on the sensitivity of birefringence and refractive index sensing are studied in this paper. The simulation results show that when the height of the polishing surface from the fiber core is less than 1.5 times the duty cycle, the closer the polishing surface is to the core, the smaller the birefringence. As the polishing angle increases, the birefringence first increases and then decreases, and the refractive index sensing sensitivity decreases accordingly. When the polishing angle is 0 degrees and the refractive index ranges from 1.330 to 1.400 , the average refractive index sensitivity of the device is as high as $3\ 457.14\ \text{nm}/\text{RIU}$. In addition, we have prepared a D-type high birefringence photonic crystal fiber SPR sensor. There is a big difference between the theoretical and experimental sensitivity values. The main reasons are: 1) The polishing surface is uneven (defects caused by air holes), which makes it difficult to completely remove the debris generated during the polishing process, which will affect the sensing performance. Performance; 2) After preparing the D-type fiber sample, the fiber is not coated in time. The D-type fiber is exposed to the air for a long time, and the dust in the air will further affect the performance of the device; 3) Although every time Before the test, the sensor will be cleaned repeatedly with ethanol, but it is difficult to completely remove the refractive index matching liquid left in the PCF air hole, which will affect the accuracy of the subsequent measurement results. For example, we tested the refractive index of 1.33 for the first time and cleaned it with alcohol. Drop 1.34 optical fiber matching liquid, because the previous liquid remains, the real value is difficult to achieve 1.34 , and the calculation is still calculated according to 1.34 ; 4) The theoretical maximum value is under the condition that the

polishing plane is parallel to the connection line of the two large air holes. The D-type high birefringence photonic crystal fiber SPR sensor was further used to test the concentration of glucose dissolved. The concentration of glucose solution increases from 0 g/dL to 10 g/dL in steps of 2 g/dL. As the glucose concentration increases, the D-type high birefringence photonic crystal fiber SPR sensor The peak wavelength of the pit of the transmission spectrum will be red-shifted. In the 0 g/dL glucose solution, the SPR resonance wavelength appeared at 578.96 nm, and when the glucose solution concentration was 10 g/dL, the SPR resonance wavelength drifted to 587.49 nm. According to the relationship between the glucose concentration and the peak wavelength of the pit of the transmission spectrum, the average sensitivity is 1.89 nm/(g/dL). The research results show that the D-shaped photonic crystal fiber SPR sensor can be applied to the fields of biology, chemistry and environmental monitoring.

Key words: Photonic crystal fiber; Surface plasmon resonance; Refractive index; Sensor; Side polishing

OCIS codes: 060.5295; 240.6680; 280.1415; 120.4640

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