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基于 PVA/Fe₃O₄ 水凝胶的锥形光纤磁传感

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摘 要: 为了研究磁性水凝胶的磁致折射率变化及其在磁致光传感领域的应用, 通过共混法制备了聚乙烯醇/四氧化三铁(PVA/Fe₃O₄)磁性水凝胶, 并基于光纤端面反射法测试了该磁性水凝胶在不同外加磁场下的折射率变化, 测得其磁致光折射率变化规律。在此基础上设计了磁性水凝胶锥形光纤传感结构。实验表明, 在环境温度 22 °C, 磁粒子浓度 2.1% 时, 6.4~22.6 mT 范围内基于磁性水凝胶的光纤磁场传感元件波长偏移灵敏度为 86.42 pm/mT; 磁粒子浓度 2.9% 时, 5.5~30 mT 范围内该传感元件波长偏移灵敏度为 51.42 pm/mT。该类磁性水凝胶在光纤磁传感测量方面具有良好的应用价值。

关键词: 光纤传感; 磁传感; 折射率; 磁性水凝胶; 锥形光纤

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0 引言

在工业生产、航天航空、电力系统等领域, 磁场对仪器设备的正常运行有很大的影响, 及时监测磁场强度对保障设备稳定运行有重要意义。光纤磁场传感器因具有安全性好、灵敏度高、响应快、体积小以及抗电磁干扰能力强等优点而被广泛应用在磁场传感领域。光纤与磁敏材料结合, 可将外界磁场的变化转换为光纤中光的波长、频率等可监测的参量变化, 实现对磁场的测量。当前, 磁流体(Magnetic fluid, MF)由于其折射率可调特性, 作为磁敏材料被广泛地应用于光纤磁场传感领域^[1-2]。ZHENG Y Z 等^[3]提出了基于微纤维模式干涉仪的磁流体磁场传感器, 磁场的变化引起磁流体折射率变化^[4-5], 使得光纤输出光的中心波长偏移, 实现磁场的传感。该类传感器具有灵敏高、质量轻、可远程探测等优点, 但其磁场测量范围受到磁流体的饱和和磁化强度影响, 高磁场感测范围有限。基于磁流体的光纤磁场传感器件通常在 0~30 mT 强度范围内探测。但工业生产中电解铝工艺会产生较高磁场, 有可能达到 50 mT, 在无人环境中远程探测时需要传感器具有较好的响应灵敏度, 并具备较大的线性响应区间。

水凝胶以水为介质, 通过共价键、范德华力或氢键等与亲水基团互相作用交联形成三维网状结构^[6]。在水凝胶基质中针对性添加有机或无机材料, 可以制备功能型复合水凝胶^[7]。与磁流体不同, 磁性水凝胶(Magnetic Hydrogel, MH)由水凝胶基质和磁性纳米粒子组成^[8-10]。Fe₃O₄ 磁粒子^[11]具有生物相容性、可控表面积、无毒无害等优点, 被用作磁性组分。WANG Yifan 等利用原位共沉淀法制备了聚乙烯亚胺(Polyethylenimine, PEI)改性壳聚糖磁性水凝胶, 应用于污水中重金属离子的吸附^[12]。BARBUCCI R 将 CoFe₂O₄ 纳米粒子共价结合到羧甲基纤维素聚合物上, 研究其流变特性^[13]。AREAL M P 通过冻融法制备了磁性聚乙烯醇(Poly(vinyl alcohol), PVA)水凝胶, 发现磁性水凝胶拥有超顺磁性^[14]。王光星等以纳米 Fe₃O₄ 和聚丙烯为原料制备了结构均匀的磁响应自修复水凝胶, 该类磁性水凝胶对磁场响应灵敏, 磁饱和强度较

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高,磁响应性强^[15]。当前研究主要关注水凝胶的磁致伸缩等磁场特性,未见对其磁致折射率变化及其在磁致光传感领域的应用。

本文提出将磁性纳米粒子基液更换为水凝胶材料,制备磁性水凝胶,寻求更高的饱和磁化强度,进一步提高光纤传感器的磁场测量范围。利用光纤端面后向反射的方法测量了制备的PVA/Fe₃O₄磁性水凝胶在不同磁场下折射率的变化,且将其包覆在锥形光纤上,利用锥形光纤的倏逝波效应^[16],测量其在磁场中的传感范围及灵敏度。

1 PVA/Fe₃O₄磁性水凝胶的制备与折射率测试

1.1 PVA/Fe₃O₄磁性水凝胶的制备

磁性水凝胶的力学和磁学性质,主要受到水凝胶材料类型、磁性粒子尺寸和浓度、水凝胶中磁性粒子的分布等的影响^[17]。根据交联机理,磁性水凝胶的制备可以选择原位沉淀法、共混法、接枝法三种^[18]。用油酸钠作为分散剂,利用共混法制备PVA/Fe₃O₄磁性水凝胶。

称取9 g聚乙烯醇(PVA,醇解度97.5%~99.0%,上海阿拉丁生化科技股份有限公司)于烧杯中,加入45 g去离子水,在磁力搅拌器(H01-1D,梅颖溥仪器仪表制造有限公司)中恒温(80 °C)搅拌30 min至PVA粉末全部溶解,烧杯内呈浓稠的液态,静置1 h消除气泡,得到PVA水凝胶基质溶液。

取3 g油酸钠(纯度>97%,上海阿拉丁生化科技股份有限公司)粉末置于30 mL去离子水中,用机械搅拌器(JB-80SH,上海析牛莱伯仪器有限公司)升温搅拌,至烧杯内呈淡黄色透明溶液,将粒径为20 nm的Fe₃O₄粒子(纯度99%,上海阿拉丁生化科技股份有限公司)注入烧杯内,剧烈搅拌30 min,静置降温后,用强磁铁吸附Fe₃O₄于烧杯底部,倾倒上层液体。同样的方法用无水乙醇清洗几次至上层液体澄清透明。通过油酸钠修饰可以使Fe₃O₄纳米粒子更好地分散于PVA水凝胶基质中。

将不同质量的磁粒子加入至相同质量的PVA水凝胶基质溶液中,超声振荡1.5 h使Fe₃O₄纳米粒子均匀分散在PVA基质溶液,可得到不同Fe₃O₄磁粒子浓度的磁性水凝胶前聚体溶液。将磁性水凝胶前聚体溶液通过毛细作用注入毛细管内,用UV胶紫外固化封装两端,置于-20 °C冰箱冷冻24 h后取出。制成冻融一次的PVA/Fe₃O₄磁性水凝胶。

1.2 PVA/Fe₃O₄水凝胶磁致折射率的测试

由于磁性水凝胶具有超顺磁性,因此磁性水凝胶遵守一般顺磁性物质材料的基本理论,可以用朗之万经典理论来研究其折射率与磁场的关系,其折射率 n_{MH} 可用朗之万函数表示^[19],即

$$n_{MH} = (n_s - n_0) \left[\coth \left(\alpha \frac{H - H_{c,n}}{T} - \frac{T}{\alpha(H - H_{c,n})} \right) \right] + n_0 (H > H_{c,n}) \quad (1)$$

式中, n_s 表示磁性水凝胶不再受磁场调制时的饱和折射率; n_0 表示当外界磁场小于 $H_{c,n}$ 时磁性水凝胶的折射率; $H_{c,n}$ 表示临界磁场,当外加磁场强度 H 大于 $H_{c,n}$ 时,磁性水凝胶折射率 n_{MH} 发生变化; α 表示拟合系数; T 表示外界环境温度。

从式(1)可以看出,对于给定浓度的磁性水凝胶,折射率受温度和磁场的影响。为定量研究磁场对磁性水凝胶折射率的影响,实验设定在22 °C的环境中进行。基于光纤端面反射的折射率测量法^[20]如图1所示。系统由宽带光源(Broadband Light Source, BBS)、光纤耦合器(Optical Fiber Coupler, OFC)和光功率计(Optical Power Meter, OPM)组成。光源连接到2×2光纤耦合器的一个耦合臂上,光功率计置于同侧的另一个耦合臂上。输出端的两根耦合臂,一个置于空气中,另一个作为测试臂与插在磁性水凝胶中的光纤跳线连接。通过改变永磁铁(Magnet)的距离改变施加到磁性水凝胶上的磁场强度,并用高斯计(Gaussmeter)监测。改变磁场强度,将测试臂光纤尖端浸入空气、水和装有磁性水凝胶的毛细管(Capillary)中,在每个条件下记录反射光的功率,并计算出磁性水凝胶折射率。实验中水、空气、光纤纤芯三种介质的折射率分别取 $n_{water} = 1.32191$ 、 $n_{air} = 1.00027$ 、 $n_{FC} = 1.4681$ 。

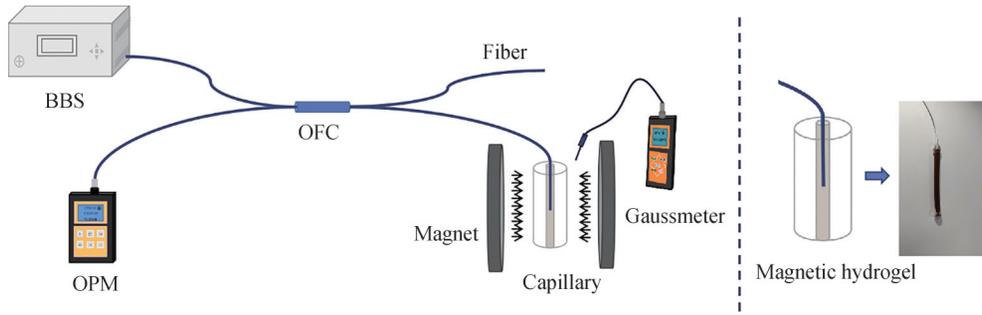


图1 磁性水凝胶折射率测试装置示意图

Fig. 1 Schematic diagram of the refractive index test device for a magnetic hydrogel

1.3 PVA/Fe₃O₄磁性水凝胶折射率分析

实验制备了浓度分别为2.1%、2.9%、3.5%的三种PVA/Fe₃O₄磁性水凝胶,测试分析三种浓度下磁性水凝胶的折射率及饱和磁场强度。图2测试结果表明,磁性水凝胶的折射率在外加磁场作用下发生明显的变化。图2(a)中,引起折射率发生变化的磁场强度范围约为5~20 mT,而浓度为2.9%时的范围约为6~30 mT,浓度为3.5%时的范围约为8~40 mT。由此可以看出,引起折射率变化的磁场强度范围随磁纳米粒子浓度的增大而增大。当磁性水凝胶置于磁场中时,磁场与磁性粒子之间及磁性粒子与磁性粒子之间产生相互作用力。由于聚合物交联网络具有变形性,凝胶网络局部结构受力发生变化,最终表现为磁性水凝胶折射率的变化。

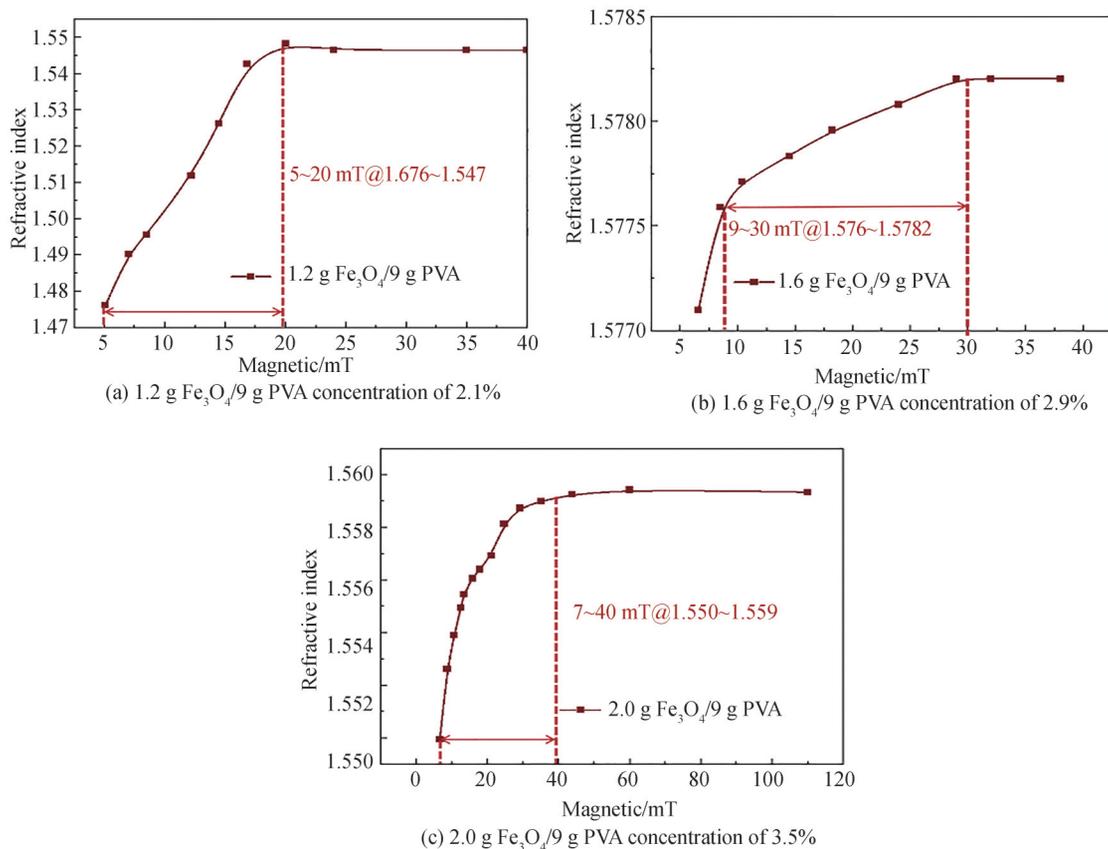


图2 不同浓度磁性水凝胶折射率随磁场变化

Fig. 2 Schematic diagram of the refractive index of magnetic hydrogels of different concentrations with magnetic field

在折射率变化响应区,随着磁粒子浓度的升高,磁性水凝胶磁饱和值增大,具备磁致折射率变化的磁场范围增大。另外,磁场的有效范围可能存在上下极值。因为过高的磁粒子浓度会导致磁粒子间距离过小,搅拌振荡过程中出现团聚现象,凝胶网格拉伸性能降低,折射率变化磁场范围减小;过低的浓度导致磁粒子

距离过大,磁粒子与磁粒子间的相互作用力减小,磁响应性能过低,磁致折射率变化不明显。

2 PVA/Fe₃O₄水凝胶的磁传感特性

2.1 水凝胶锥形光纤磁传感设计

光纤由纤芯和包层组成,两者折射率不同,光纤中绝大部分的光被限制在纤芯中传播,仅有少许光在包层中传播,形成倏逝波。当倏逝波在包层中传输一段距离后,能量会衰减到纤芯和包层分界面能量的1/e,此时的传输距离被称为透射深度,可用 d_p 表示,即

$$d_p = \frac{\lambda}{2\pi} \frac{1}{\sqrt{n_1^2 \sin^2 \theta - n_2^2}} \quad (2)$$

式中, λ 为入射光波长, θ 为纤芯和包层界面的入射角度, n_1 和 n_2 分别为纤芯和包层的折射率。

光纤本身的损耗很低,透射深度远小于包层厚度,因此不受外界环境折射率的影响。锥形光纤有效减小光纤直径,使倏逝波与外界环境物质直接接触,可实现对外界环境的传感。实验所设计的是具有两个锥形过渡区和一个均匀细腰区的锥形光纤,如图3(b)所示。

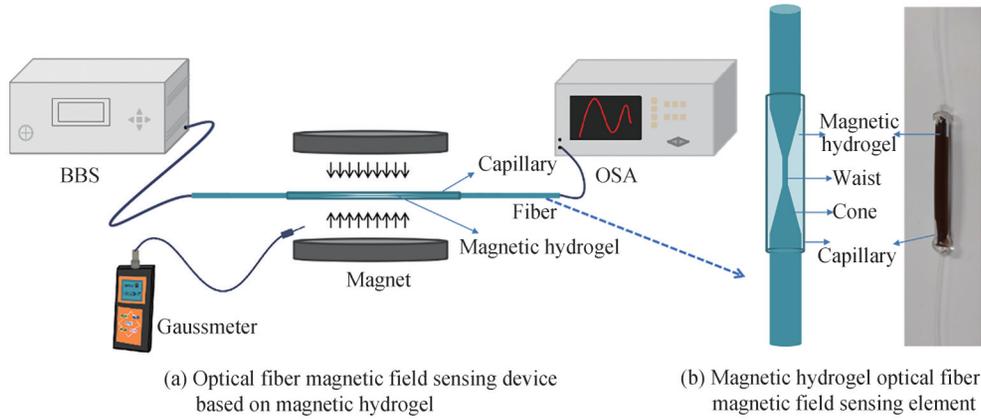


图3 基于磁性水凝胶的光纤磁场传感实验装置

Fig. 3 Diagram of optical fiber magnetic field sensing device based on magnetic hydrogel

在锥形过渡区,一部分基模 HE_{11} 的能量耦合进入包层模 HE_{1m} 中,其本质上也是一种模式干涉,其产生的干涉光谱强度可以用公式表示为

$$I_1 = I_1 + I_2 + 2\sqrt{I_1 I_2} \cos \varphi \quad (3)$$

式中, I_1 为基模的光强, I_2 为包层模的光强, φ 为基模与包层模间的相位差, $\varphi = 2\pi(n_{\text{eff},1} - n_{\text{eff},2})L/\lambda$, $(n_{\text{eff},1} - n_{\text{eff},2})$ 是基模和包层模有效折射率之差, L 为传感区域的有效长度。

根据式(3),当 $\varphi = (2m + 1)\pi$ (m 为一个整数)时,干涉强度取最大或最小,此时对应的波长为

$$\lambda = \frac{2\Delta n_{\text{eff}} L}{2m + 1} \quad (4)$$

由此可推出两个相邻干涉峰的波长间隔,即自由光谱范围(FSR)为

$$\text{FSR} = \frac{4\Delta n_{\text{eff}} L}{(2m - 1)(2m + 1)} \approx \frac{\lambda^2}{\Delta n_{\text{eff}} L} \quad (5)$$

环境折射率的改变会引起光纤传输模式的有效折射率改变,进而引起有效折射率之差 Δn_{eff} 改变。根据式(5)可知,干涉谷波长会发生变化,可以推导出干涉谷波长随环境折射率改变的表达式为

$$\Delta \lambda = \frac{2(\Delta n_{\text{eff}} + \Delta \lambda) L}{2m + 1} - \frac{2\Delta n_{\text{eff}} L}{2m + 1} = \frac{2\Delta n L}{2m + 1} \quad (6)$$

基于磁性水凝胶的锥形光纤传感器,利用磁性水凝胶在磁场作用下折射率改变的特性,通过测量干涉谷波长的漂移量获得磁场信息。

实验利用光纤熔接机(Vytran,GPX-3000),将单模光纤(SMF-28e+)拉制成锥区长度为3 mm,腰区长度为6 mm,直径为11.8 μm的锥形光纤。直径为3 mm的毛细管包覆在锥形光纤锥区和腰区处,然后将PVA/Fe₃O₄磁性水凝胶前聚体溶液填充入毛细管中,并用UV胶固化毛细管两端。将其置于-20℃冰箱冷冻24 h后取出,室温下放置2 h。所得的锥形光纤传感结构如图3所示。

将光纤传感元两端分别接入光纤宽带光源(脉锐光电,ASE)及OSA光谱仪(Yokogawa,AQ6370),锥形光纤结构置于22℃恒定温度下的磁场中。通过改变两磁铁间的距离改变施加在传感元的磁场强度,通过检测输出光谱的波长漂移来实现磁场的探测。

2.2 磁传感结果分析

实验制备了两种Fe₃O₄浓度的PVA/Fe₃O₄磁性水凝胶,分别为2.1%磁粒子浓度(9 g PVA/45 g H₂O水凝胶基质加入1.2 g Fe₃O₄)和2.9%磁粒子浓度(9 g PVA/45 g H₂O水凝胶基质加入1.6 g Fe₃O₄)。锥形光纤放置于可调强度磁场中,测得透射光谱如图4和图5所示。

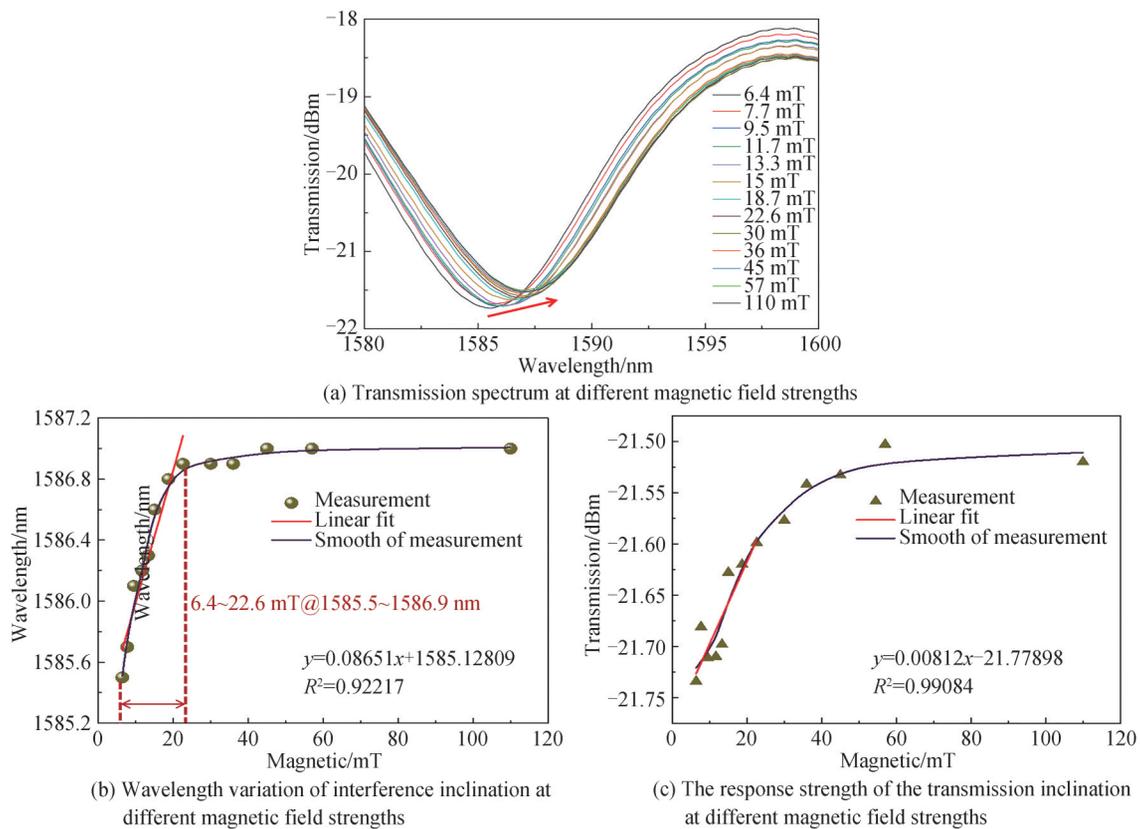


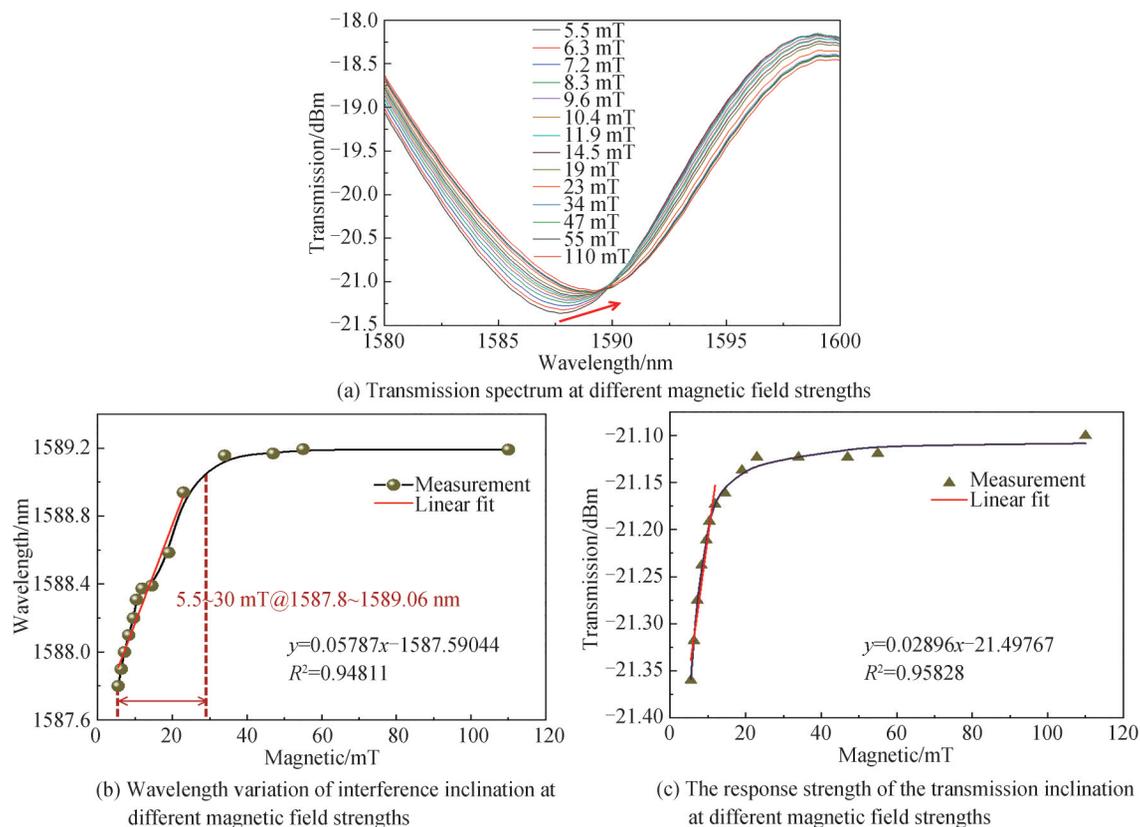
图4 1.2 g Fe₃O₄/9 g PVA浓度的磁性水凝胶包覆锥形光纤实验结果

Fig. 4 Experimental results of magnetic hydrogel-coated conical fiber at 1.2 g Fe₃O₄/9 g PVA concentration

从图4(a)看出,穿过锥形光纤的宽带光谱,透射率极小值(波谷)处的波长发生红移;且随着外加磁场强度的增加,波谷处的透射强度也同时增大。图4(b)为波谷波长随外加磁场强度变化的波长偏移曲线。当磁场强度从6.4 mT逐步增加到22.6 mT时,波谷从1585.5 nm相应移动到1586.9 nm。随后磁致波长偏移变化平缓,可以认为磁性水凝胶逐渐达到饱和磁化强度(约22.6 mT)。6.4~22.6 mT范围内波长偏移具有线性响应的特性,波长偏移灵敏度约为86.42 pm/mT,饱和磁化强度约为30 mT。图4(c)是波谷处透射强度随磁场变化的关系。当磁场从6.4 mT增加到22.6 mT时,其波谷信号强度从-21.73 dBm增强到-21.60 dBm,在此范围内光强变化灵敏度为0.057 dBm/Oe。比较可知,通过波长偏移进行磁传感测试更方便。

比较图2(a)和图4(b)可知,在磁场响应的线性区间,PVA/Fe₃O₄磁性水凝胶的磁致折射率变化曲线,与锥形光纤的波长偏移变化曲线,变化规律一致,表明PVA/Fe₃O₄磁性水凝胶是有效的磁传感材料。

从图5看出,2.9%磁粒子浓度的传感单元,在5.5~30 mT范围内波长偏移具有线性响应特性,波长偏移

图5 1.6 g Fe₃O₄/9 g PVA 浓度的磁性水凝胶包覆锥形光纤实验结果Fig. 5 Experimental results of magnetic hydrogel-coated conical fiber at 1.6 g Fe₃O₄/9 g PVA concentration

灵敏度约为 51.42 pm/mT, 饱和磁化强度约为 40 mT。与图 4 比较可知, 当 PVA/Fe₃O₄ 磁性水凝胶浓度变大时, 磁致光波长偏移变化规律保持一致, 磁场响应范围有所提升, 饱和磁化强度变大, 但波长灵敏度有所降低。理论上, 磁性水凝胶的磁致折射率变化值随磁纳米粒子的浓度增大而增大, 传感器可测量的磁传感范围和饱和磁化强度随磁纳米粒子浓度增大而增大。但是, 增加磁粒子浓度以提升磁传感响应能力不是无限的。由于磁性水凝胶的饱和磁化效应, 基于磁性水凝胶的磁场传感器的非线性响应不可避免。当施加相对较低强度的磁场时, 磁凝胶中最初均匀分布的磁纳米粒子向磁场方向聚集, 从而改变磁性水凝胶的折射率, 当外部磁场强度接近或大于饱和磁化强度时, 大多数纳米粒子已经聚集完成, 因此折射率不再改变。

对于锥形光纤磁传感而言, 波长偏移灵敏度与线性响应范围受限于锥形结构及磁纳米粒子的浓度。首先, 优化锥区的长度和锥径等参数可以提高灵敏度^[21], 但更细更长的光纤极易断裂, 在拉锥工艺上要求更高, 且受限于设备加工能力。其次, 提高磁纳米粒子的浓度, 可以提高磁场响应线性范围, 但是更高的浓度会造成磁粒子的团聚, 甚至达到折射率变化极限。本研究在上述影响因素上做了较好的兼顾。如表 1 所示,

表 1 磁场线性响应范围及灵敏度对比

Table 1 Linear response range and sensitivity comparison of magnetic field

Magnetic particle diameter/concentration	Linear response range	Sensing structure	Sensitivity within the linear response range	Reference
10 nm/1.8%	11 mT	A tapered fiber with a diameter of 7 μm in the waist area	293 pm/Oe	[3]
10 nm/1.18 g $\cdot\text{cm}^{-3}$	10~22.5 mT	A tapered fiber with a diameter of 3.88 μm in the waist area	191.8 pm/Oe	[22]
10 nm/1.27 g $\cdot\text{cm}^{-3}$	3~9 mT	A section of four-core fiber (FCF) sandwiched between two no-core fibers (NCFs) forms an NCF-FCF-NCF structure	264.29 pm/mT	[23]
10 nm/3.6%	0.6~21.4 mT	A multimode-single mode-multimode (MM-SM-MM) fiber structure	123.06 pm/mT	[24]
20 nm/2.1%	6.4~22.6 mT	A tapered fiber with a diameter of 11.8 μm in the waist area	86.42 pm/mT	This study

与文献[3]中基于磁流体的锥形磁传感元件的结果相比,本研究在磁粒子浓度略高的情况下,基于磁性水凝胶的锥形光纤传感元件磁场线性响应范围增大到两倍;与文献[22]、[23]、[24]中不同浓度的磁流体传感元件测试结果相比,在浓度降低的情况下,线性响应范围相当。另外,本研究所拉制的锥形光纤,腰区直径11.8 μm大于文献[3]、[22]采用的3.88 μm及7 μm,同时实现了更大的灵敏度,降低了拉制锥形光纤的工艺要求。

3 结论

本文制备了一种新型PVA/Fe₃O₄磁性水凝胶,测试发现施加磁场激励下PVA/Fe₃O₄水凝胶拥有明显的磁致折射率变化特性。基于PVA/Fe₃O₄水凝胶制备的锥形光纤磁传感元件,水凝胶掺杂磁粒子浓度为2.1%时,外加磁场在6.4~22.6 mT范围内,光谱仪测得的波长响应灵敏度达到86.42 pm/mT;磁粒子浓度为2.9%时,5.5~30 mT磁场范围内波长响应灵敏度达到51.42 pm/mT。在较低的磁粒子浓度、锥形光纤直径宽容的情况下,锥形光纤磁传感元件表现出良好的线性磁场响应范围及灵敏度。研究表明磁性水凝胶在光纤磁传感测量方面有应用价值。通过提高磁性纳米粒子在水凝胶中的分散均匀度,优化锥形光纤传感结构,设计干涉调制光路结构等工作,可以进一步提高传感器性能。

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Magnetic Field Sensing of Conical Optical Fibers Based on PVA/Fe₃O₄ Hydrogel

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Abstract: Magnetic fluids are widely used in the field of optical fiber magnetic field sensing as magnetic sensitive materials due to the adjustable refractive index. Optical fiber magnetic field sensor based on magnetic fluid has the advantages of high sensitivity and light weight, but the magnetic field measurement range of this kind of sensor is affected by the saturation magnetization of magnetic fluid, which limits the high magnetic field sensing range. Some studies have shown that magnetic hydrogels have the characteristics of high sensitivity to magnetic field, high saturation magnetization, high magnetic responsiveness and super magnetic similarity with magnetic fluids. In this paper, it is proposed to replace the base liquid of magnetic nanoparticles of magnetic fluid with hydrogel material to prepare magnetic hydrogel, hoping to seek a higher level of saturation magnetization, and make prospects for further improving the magnetic field measurement range of optical fiber sensors. A polyvinyl alcohol/ferric oxide (PVA/Fe₃O₄) magnetic hydrogel was prepared by the composite method, and used a fiber end face reflection-based measurement method to test the refractive index variation of the magnetic hydrogel under different magnetic fields, which proved that it had magnetic-induced refractive index variation characteristics and could be applied in the fiber magnetic field sensing structure based on refractive index variation. Based on this research, a cone-shaped fiber sensing structure based on magnetic hydrogel was designed. Single-mode optical fiber is pulled into a cone shape by melting and pulling method, and magnetic hydrogel is wrapped in the cone area and waist area of the cone-shaped optical fiber. The input

light in the fiber will generate evanescent wave when it is in the conical transition region, which will excite the higher-order cladding mode in the fiber. After the fundamental mode and cladding mode are transmitted in the waist region of the tapered fiber, mode coupling and mode interference will occur when entering another tapered region. When the intensity of the magnetic field applied in the external environment changes, the refractive index of the magnetic hydrogel will change. In this experiment, we change the refractive index of the magnetic hydrogel by applying magnetic fields of different intensities to the sensing element of the tapered fiber wrapped by the magnetic hydrogel. The spectral change of the output light of the tapered fiber is detected by using the evanescent wave in the tapered region of the tapered fiber, which is sensitive to the refractive index of the external environment, to study the magnetic field sensing characteristics of this structure. The experimental results show that the sensitivity of wavelength shift is 86.42 pm/mT within the range of 6.4~22.6 mT, when the magnetic particle concentration is 2.1% at a constant temperature of 22 °C. And also the sensitivity of wavelength shift is 51.42 pm/mT within the range of 5.5~30 mT, when the magnetic particle concentration is 2.9%. From the point of view of optical fiber magnetic sensing measurement, magnetic hydrogel has high application value and deserves further research. This paper also proposes that the detection sensitivity can be improved by selecting more appropriate dispersants to improve the dispersion uniformity of magnetic nanoparticles in hydrogels, and by improving the structure of optical fiber sensing elements. During the preparation of magnetic hydrogel, we can further improve the magnetic field sensing range and expand the application of magnetic hydrogel in the field of optical fiber magnetic field sensing by optimizing the freezing and thawing times and the concentration of magnetic nanoparticles.

Key words: Fiber optic sensing; Magnetic field sensing; Refractive index; Magnetic hydrogel; Conical optical fiber

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