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基于PVA涂覆的U型超细光纤湿度传感器

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摘　要：提出了一种基于聚乙烯醇(PVA)涂覆的U型超细光纤湿度传感器。光纤熔融拉锥机将单模光纤拉制成微米级的超细光纤，采用滴涂法将PVA溶液均匀的涂覆在光纤表面。为了减小传感器体积使其便于测量，将超细光纤固定为U型。实验发现，未涂覆PVA时，U型传感器的湿度灵敏度极低；涂覆PVA后，传感器在34%RH～90%RH的检测范围内湿度灵敏度高达146.1 pm/%RH。温度实验表明，传感器在40 ℃～80 ℃范围内温度灵敏度为15.8 pm/℃。该传感器制备过程简单、灵敏度高、便于携带、成本低、温度串扰影响较小，在湿度检测领域有广泛的应用前景。

关键词：超细光纤；U型；聚乙烯醇；温度；湿度
中图分类号：TN253　　**文献标识码**：A　　**doi**：10.3788/gzxb20235212.1206002

0　引言

湿度是传感领域的重要参数之一，湿度的监测与控制应用于许多场合，如农业[1]、电力[2]、环境安全[3]、医药工程[4]等。与传统湿度传感器相比，光纤湿度传感器具有耐腐蚀、重量轻、可远程操作、不受电磁干扰等优点，近年来得到了广泛研究。目前，根据加工方案的不同，已经制备出了各种不同的光纤湿度传感器，如：长周期光纤光栅[5]，Sagnac环[6]，马赫-曾德干涉仪[7]，布拉格光栅[8]，Fabry-Perot腔[9]、迈克尔逊干涉仪[10]等。

由于U型超细光纤具有体积小、制作成本低、灵敏度高等优点，受到各界广泛关注。2020年，XU Yue等设计了一款快速响应的U型微光纤应变传感器[11]。该传感器将U形超细纤维嵌入聚二甲基硅氧烷(Polydimethylsiloxane，PDMS)薄膜中来检测微量流量。当PDMS薄膜浸入液体时，流动的液体会导致嵌入的超细纤维的透光率发生变化，这种变化与液体的流速直接相关。该传感器的分辨率为0.005 L/min，检出限为0.05 L/min。2022年，SUN Dandan等设计了一种涂有二硫化钼薄膜的U型微光纤干涉仪[12]。当外界环境中湿度和温度变化时，光纤表面的二硫化钼导致传感器折射率发生变化，干涉仪的波长因此发生漂移。结果表明，该传感器在51%RH～80%RH下的相对湿度灵敏度0.116 nm/%RH。因为超细光纤本身对湿度不敏感，为达到检测湿度的目的，大多数光纤湿度传感器需要在光纤表面涂覆一层湿敏材料。常见的湿敏材料有：明胶[13]、聚乙烯醇[14]、氧化石墨烯[15]、聚酰亚胺[16]、有机金属骨架[17]等。其中聚乙烯醇(Polyvinyl Alcohol，PVA)作为一种强亲水性材料，吸水后体积会增大，其折射率会随周围湿度变化而变化，将PVA均匀的涂覆在U型超细光纤表面可以检测湿度[18-19]。

本文对未涂覆PVA的U型传感器(S-1)和涂覆PVA的U型传感器(S-2)进行了湿度测试。实验结果发现，由于PVA覆盖层的存在，使得传感器对周围环境湿度的变化非常敏感。在室温28 ℃下，该传感器在34%RH～90%RH的检测范围内平均湿度灵敏度高达146.1 pm/%RH。该传感器稳定性良好，在湿度增减过程中具有较高的可逆性，温度串扰影响较小，制备方法简单、成本低、便于携带，具有广阔的应用前景。

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1 传感器制作及理论分析

1.1 传感器传感原理分析

PVA是一种强亲水性材料，其折射率随周围湿度变化而发生改变。由于PVA在二氧化硅表面具有高附着力，因此可以较容易地涂覆在光纤表面。因为PVA的这些特殊性能，将PVA与超细光纤结合，可以用来测量湿度。PVA的厚度会对传感器的光耦合损耗和相对湿度灵敏度等性能产生重要影响。增厚PVA涂层，会同时增加传感器的损耗和相对湿度灵敏度。因此，需要选择合适的PVA涂层来优化湿度传感器的性能。本文分别制作了三种涂层厚度的传感器，来探索其对传感器的影响。

当光纤逐渐变细，使其腰径接近或小于光的波长时，光纤就变成了一个带有空气包层的波导。因此，部分光可以在空气包层中传播，从而使周围环境与光之间产生强烈而快速的近场相互作用[19]。当光从光源进入U型传感器的弯曲过渡区时，高阶模式(LP_{1n})被激活。高阶模通过腰区后在过渡区与基模(LP_{01})结合，由于基模(LP_{01})与高阶模(LP_{1n})的有效折射率不同，因此会产生干涉。基模(LP_{01})和高阶模(LP_{11})具有相似的方位角对称性和最小的相位失配，因此耦合主要发生在基模(LP_{01})和高阶模(LP_{11})之间[20]。为了方便计算，设LP_{01}模式和LP_{11}模式的有效折射率分别为$n_{\mathrm{eff}}^{\mathrm{LP}_{01}}$和$n_{\mathrm{eff}}^{\mathrm{LP}_{11}}$。因此，传感器的透射强度($T$)可以表述为

$$T = T_1 + T_2 + 2\sqrt{T_1 T_2}\cos\delta\phi \tag{1}$$

式中，T_1表示LP_{01}模式的功率，T_2表示LP_{11}模式的功率，$\delta\phi$是两种模式的累积相位差，表示为

$$\delta\phi = 2\pi\Delta nL/\lambda \tag{2}$$

式中，$\Delta n = n_{\mathrm{eff}}^{\mathrm{LP}_{01}} - n_{\mathrm{eff}}^{\mathrm{LP}_{11}}$是有效折射率差，$\Delta n$对外界环境的湿度变化非常敏感；$L$为传感器的有效物理长度；$\lambda$是光源的中心波长。为了更进一步理解外部环境RI的光谱响应特性，将式(2)中的$\delta\phi$视为常数，得到

$$\frac{\mathrm{d}\lambda}{\mathrm{dRH}} = \frac{\lambda}{\Delta n_{\mathrm{eff}}}\left(\frac{\partial n_{\mathrm{eff}}^{\mathrm{LP}_{01}}}{\partial n_{\mathrm{air}}}\frac{\mathrm{d}n_{\mathrm{air}}}{\mathrm{dRH}} - \frac{\partial n_{\mathrm{eff}}^{\mathrm{LP}_{11}}}{\partial n_{\mathrm{air}}}\frac{\mathrm{d}n_{\mathrm{air}}}{\mathrm{dRH}}\right)\Bigg/\left[1 - \frac{\lambda}{\Delta n_{\mathrm{eff}}}\left(\frac{\partial n_{\mathrm{eff}}^{\mathrm{LP}_{01}}}{\partial\lambda} - \frac{\partial n_{\mathrm{eff}}^{\mathrm{LP}_{11}}}{\partial\lambda}\right)\right] \tag{3}$$

式中，n_{air}是周围空气的折射率，而$1 - \frac{\lambda}{\Delta n_{\mathrm{eff}}}\left(\frac{\partial n_{\mathrm{eff}}^{\mathrm{LP}_{01}}}{\partial\lambda} - \frac{\partial n_{\mathrm{eff}}^{\mathrm{LP}_{11}}}{\partial\lambda}\right)$是色散因子。由式(3)可知，传感器的灵敏度主要由模态的有效折射率变化以及色散因子决定。由于超细光纤提供了强大的倏逝场，当其与周围环境相互作用时，随着环境湿度的增加，n_{air}也会增加，LP_{01}和LP_{11}模式的有效折射率也会增加。因为各模态的倏逝场能量占比不同，所以两种模态的折射率增加程度不同。在本文设计的基于PVA的U型超细光纤传感器中，当外部环境湿度增高时，LP_{11}模式的有效折射率增量大于LP_{01}模式的有效折射率增量，可以得出$\left(\frac{\partial n_{\mathrm{eff}}^{\mathrm{LP}_{01}}}{\partial n_{\mathrm{air}}}\frac{\mathrm{d}n_{\mathrm{air}}}{\mathrm{dRH}} - \frac{\partial n_{\mathrm{eff}}^{\mathrm{LP}_{11}}}{\partial n_{\mathrm{air}}}\frac{\mathrm{d}n_{\mathrm{air}}}{\mathrm{dRH}} < 0\right)$，色散因子通常为负。因此，随着环境中相对湿度的增加，传感器的透射光谱会出现一定的红移。由此可以看出，传感器周围环境的湿度变化会导致光谱偏移，因此可以通过观察光谱的偏移来测量湿度。

1.2 传感器结构设计及制作

1.2.1 材料和试剂

本实验所使用的光纤均为单模光纤，由长飞公司生产，其纤芯直径为9 μm，包层直径125 μm。实验中用到的PVA生产厂家是麦克林公司，醇解度99%。制作PVA溶液的步骤非常简单，具体过程为：取0.25 g PVA倒入玻璃瓶内，加入4.75 mL超纯水；设置磁力搅拌器温度为80 ℃，搅拌速度800转/min，搅拌2 h；为了去除溶液中气泡等因素对实验结果的影响，PVA静置一夜后使用。

1.2.2 制备超细纤维

超细光纤由山东凯普乐有限公司生产的光纤熔融拉锥机制作完成，具体步骤为：取1 m长的单模光纤，将其对折找到中心点；在中心点处，用剥线钳刮掉光纤涂覆层，长度约2 cm；用无尘纸和酒精清洁光纤表面，防止光纤碎屑对实验结果产生影响。将光纤放置在拉锥机凹槽内，通过移动光纤，使去除涂覆层部分的单模光纤的中心位置对准火焰喷头，然后使用夹具固定；设置拉锥机参数后启动。具体拉锥过程如图1所示。

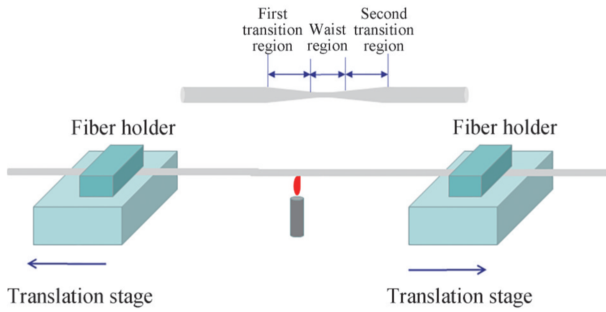


图1　超细光纤制备示意图
Fig.1　Schematic diagram of microfiber fabrication

1.2.3　制备湿度传感器

首先制备一个简易的玻璃模具用来放置超细光纤,将模具固定在步进电机上,通过步进电机带动模具中的超细光纤。用支架上的烧杯夹夹紧一根滴管,用移液枪将配置好的PVA液滴涂覆在滴管上,保证滴管上的液滴均匀、悬空。将PVA液滴缓速穿过光纤,直至液滴包裹整根光纤。这里需要特别注意液滴摆放位置,它必须放置在距离中心点二分之一的地方,这样做的目的是为了保证涂覆的对称性及均匀性,这些因素会对传感器的灵敏度、线性度产生很大影响。步进电机带动光纤,将PVA均匀的涂覆在光纤表面。具体涂覆方法如图2所示。值得一提的是,改变电机移动速度、涂覆次数等,可以改变涂层厚度,涂层厚度会影响传感器的性能。将涂覆完成的超细光纤,穿过自制的模具,模具由三根毛细玻璃管制成,通过UV光刻胶及紫外线灯,将超细光纤固定为U型。将传感器放置在真空干燥箱中,待光纤表面的PVA溶液中的溶剂挥发完后,会留下一层PVA薄膜。传感器结构如图3所示,其腰区最细部分为10.96 μm。为了观察传感器表面涂

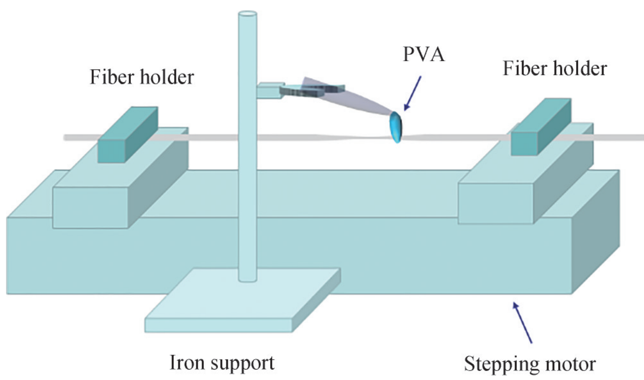


图2　涂覆PVA的过程示意图
Fig.2　Schematic diagram of the process of coated PVA

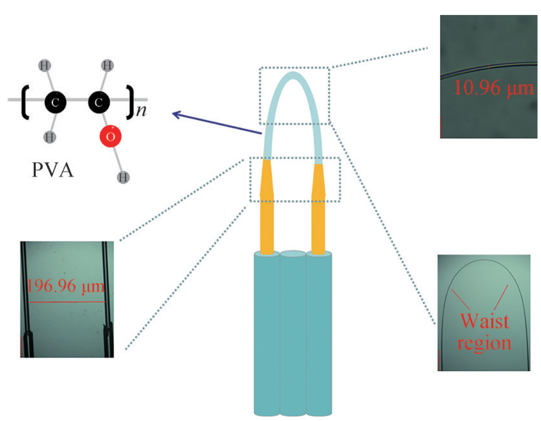


图3　U型传感器结构示意图
Fig.3　U-shaped sensor structure illustration

覆情况，将传感器放在电镜下观察，结果如图4所示，可以发现在超细光纤表面有一层PVA薄膜。图5是传感器在34%RH下的透射谱。其中，图5(a)为不涂覆PVA时的传感器(S-1)透射谱，图5(b)为涂覆后的传感器(S-2)透射谱，对比发现，涂覆PVA前后两种传感器的输出光谱略有不同。这与两种传感器的过渡区结构参数以及涂层结构有关。

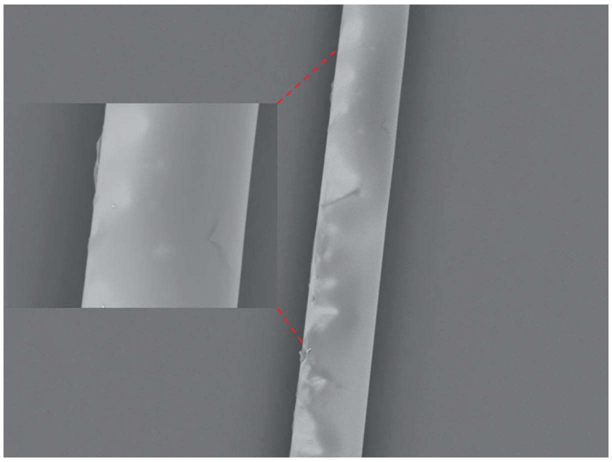
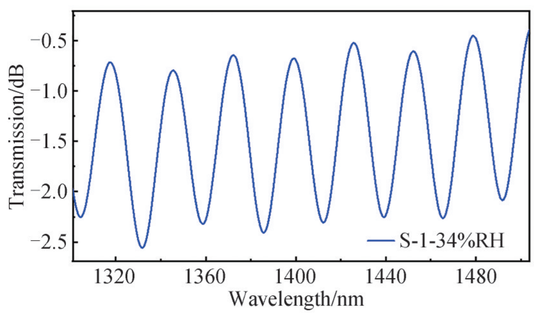
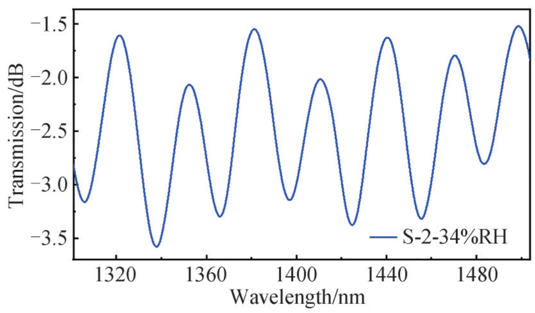


图4　U型传感器电镜图

Fig.4　U-shaped sensor electron microscope diagram



(a) Sensor S-1



(b) Sensor S-2

图5　传感器在34%RH的透射谱

Fig.5　Transmittance spectrum of the sensor at 34% RH

2　传感器湿度实验

图6是传感器湿度实验的设备图。该湿度传感器的光谱由光源、日本横河公司生产的光谱分析仪(YOKOGAWA，AQ6370D)、不同湿度的饱和盐溶液、密封湿度瓶、以及可同时测量湿度和温度的电子湿度传感器(DT-625，湿度：0～20%RH，精度为±3.5%RH；20%RH～80%RH，精度为±2.5%RH；80%RH～100%RH，精度为±3.5%RH，温度：−30～100℃，精度为±0.5℃)获得。在实验中，将U型传感器和湿度传感器固定在密封的湿度箱里，通过电子湿度传感器可以实时获得箱内湿度、温度变化。由于不同的饱和盐溶液湿度不同，通过选择合适的饱和盐溶液，可以获得合理的湿度梯度，用于不同湿度环境下的湿度测量。

实验过程中，使用单模光纤将U型传感器与光源、光谱仪相连。湿度测量在室温28℃下进行的，为尽量减小温度、气流等对实验结果的影响，所有实验均在超净间完成。选用不同的饱和盐溶液，设置了相对湿度为34%RH、46%RH、59%RH、73%RH和90%RH的密封室。首先，研究了不同涂层厚度下传感器的湿度响应能力。本文制作了三种涂层厚度的湿度传感器，分别为：不涂覆PVA的传感器S-1、涂覆三次的传感器S-2、涂覆五次的传感器S-3。在湿度从低到高的过程中，当特定湿度达到稳定值时，记录传感器的光谱图。如图7所示，为S-1传感器湿度从34%RH到90%RH升高时，谐振峰在1 420～1 475 nm处波长随湿度变化的光谱图与1 450～1 480 nm处的拟合。实验结果表明，随着湿度的增加，传感器S-1对湿度不敏感。将涂覆三次的传感器S-2置于密封的湿度室内，如图8所示，为传感器S-2在1 430～1 488 nm处的谐振峰波长随

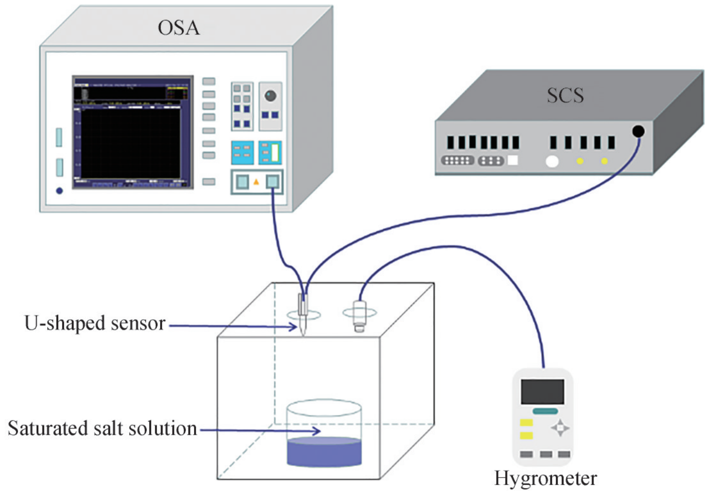


图6　传感器湿度实验设备图

Fig.6　Sensor humidity experimental equipment diagram

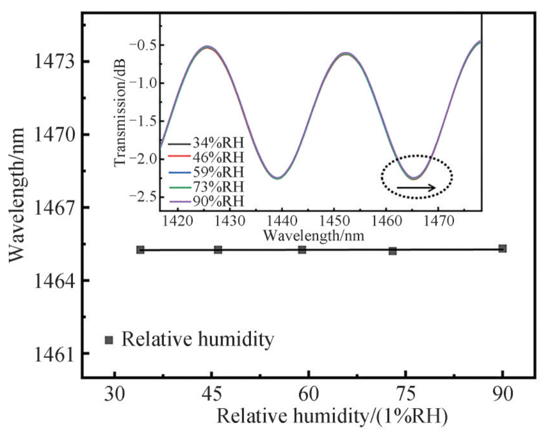


图7　传感器S-1在1 420～1 475 nm处的谐振峰波长随湿度变化的光谱与1 450～1 480 nm处的拟合

Fig.7　Spectrogram of resonant peak wavelength at 1 420～1 475 with humidity for sensor S-1 with a fit at 1 450～1 480 nm

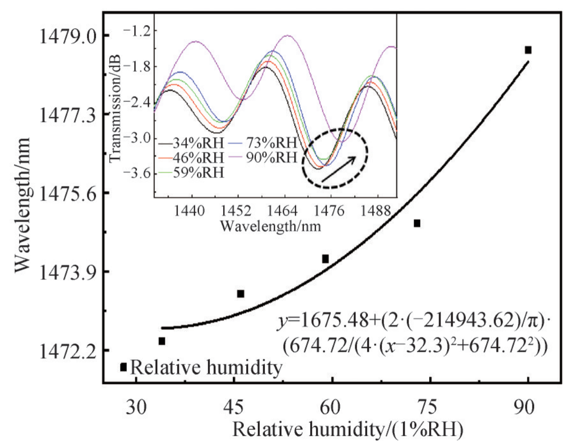


图8　传感器S-2在1 430～1 488 nm处的谐振峰波长随湿度变化的光谱与1 464～1 488 nm处的拟合

Fig.8　Spectrogram of resonant peak wavelength at 1 430～1 488 with humidity for sensor S-2 with a fit at 1 464～1 488 nm

湿度变化的光谱图与1 464～1 488 nm处的拟合，发现光谱有明显的红移，通过计算，得到传感器S-2的湿度灵敏度为112.1 pm/%RH。图9是涂覆五次的传感器S-3在1 410～1 470 nm处谐振峰波长随湿度变化的光谱图，实验结果表明，传感器S-3的湿度响应能力优于其他两根传感器。如图10所示，为传感器S-3在1 440～1 470 nm处的谐振峰波长随湿度变化的拟合。实验结果表明，S-3传感器的中心波长随环境湿度增加而增加，随环境湿度降低而减小，它们的拟合程度R^2分别为0.994 09和0.991 54，拟合程度较高，上升和下降的拟合曲线几乎一致，能够满足湿度测量需求。湿度增加时，传感器的灵敏度约为146.1 pm/%RH，湿度下降时，传感器的灵敏度约为140.9 pm/%RH。在湿度增加和减少的过程中，传感器的灵敏度有轻微的不同。这可能是PVA膜吸收的水分子没有全部释放到周围环境中导致的，通过延长实验时间可以尽可能地减小该现象。因此，该传感器S-3具有较高的灵敏度及增减过程的可逆性，传感器的最佳检测范围为34%RH～90%RH。通过对比三根传感器，发现：增加涂层厚度，可以增加传感器的灵敏度；但是涂层越厚，传感器的损耗越大，且随着涂层厚度的增加，会导致传感器的制作时间变长，这会使滴管上的PVA液滴挥发，从而影响涂覆的均匀性。因此，需要选择合适的涂层厚度。

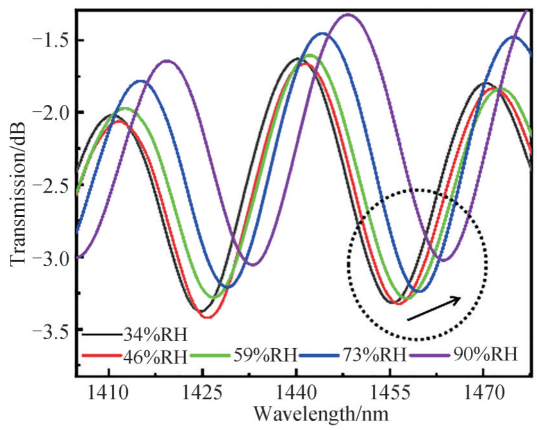


图 9　传感器 S-3 在 1 410～1 470 处的谐振峰波长随湿度变化的光谱

Fig.9　Spectral diagram of the resonant peak wavelength of sensor S-3 at 1 410～1 470 nm as a function of humidity

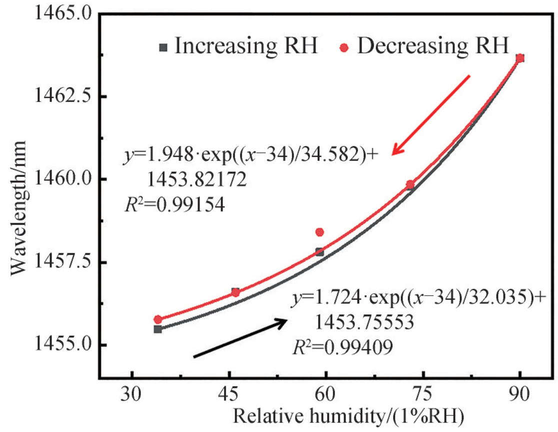


图 10　传感器 S-3 在 1 440～1 470 处的谐振峰波长随湿度变化的拟合

Fig.10　Fitting diagram of resonant peak wavelength variation with humidity of sensor S-3 at 1 440～1 470 nm

为了探究超细光纤腰区直径对传感器湿度灵敏度的影响，制作了腰区直径 12.41 μm 的传感器 S-4，其腰区直径比传感器 S-3（腰区直径：9.84 μm）更大。采用与传感器 S-3 同样的方法，在传感器 S-4 表面涂覆 5 次 PVA。如图 11 所示，为传感器 S-4 在 1 411～1 485 nm 处的谐振峰波长随湿度变化的光谱与 1 462～1 485 nm 处的拟合，其湿度灵敏度为 101.2 pm/%RH。与 S-4 传感器相比，S-3 传感器明显具有更高的灵敏度。这是因为腰径越小的超细光纤与外界环境耦合的倏逝场越强，从而加强了它们之间的相互作用，减少了色散[21]。为了更直观的比较四种传感器的性能，将实验结果绘制成表格，如表 1 所示。

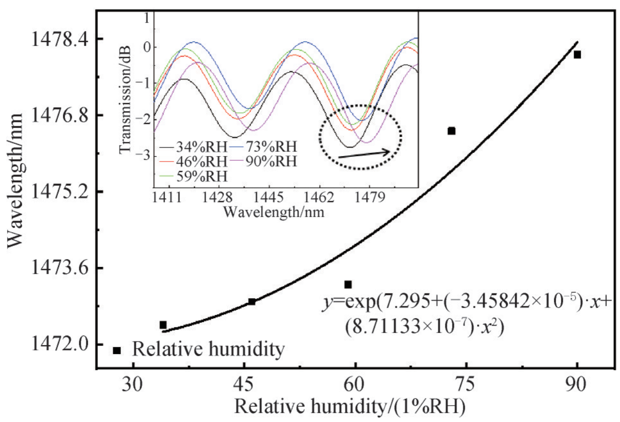


图 11　传感器 S-4 在 1 411～1 485 nm 处的谐振峰波长随湿度变化的光谱与 1 462～1 485 nm 处的拟合

Fig.11　Spectrogram of resonant peak wavelength at 1 411～1 485 nm with humidity for sensor S-4 with a fit at 1 462～1 485 nm

表 1　传感器相关参数

Table 1　Related parameters of the sensor

Sample	Number of coats	Waist diameter/μm	Humidity sensitivity/(pm/%RH)
Sensor 1	0	9.08	≈0
Sensor 2	3	9.68	112.1
Sensor 3	5	9.84	146.1
Sensor 4	5	12.41	101.2

为了进一步检验传感器的稳定性，将传感器 S-3 放置在密封的湿度室中，湿度分别为 46%RH 和 73%RH。每 10 min 记录一次湿度的光谱，实验结果如图 12 所示，在 60 min 内，传感器中心波 1 456.4 nm、1 459.7 nm 处的峰值几乎不发生漂移，这说明该传感器具有良好的稳定性。实验出现的偏差可能是由湿度瓶内的循环气流和记录时的机械振动、误差等引起的。

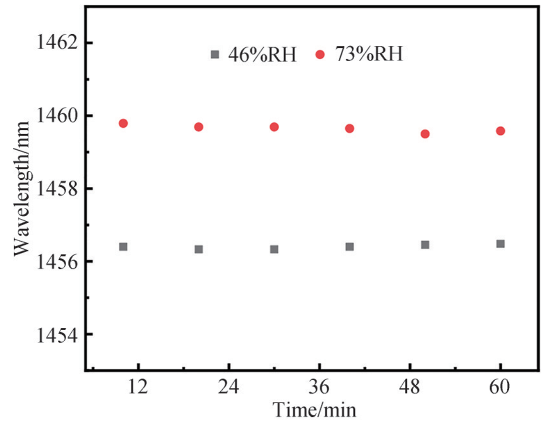


图12　传感器S-2湿度稳定性示意图

Fig.12　Diagram of sensor S-2 humidity stability

3　传感器温度实验

在湿度测量实验中,温度的波动对实验结果产生一定影响。这是因为温度会改变PVA薄膜和U型超细光纤的理化性,如折射率和膨胀度的变化。因此,探究温度对传感器的串扰影响是十分必要的。

将样品S-3固定在恒温加热平台(JF-956S)上,实验过程中保持环境湿度不变,控制加热平台温度从40 ℃均匀上升到80 ℃,每隔10 ℃记录一次传感器的光谱图。当加热台的温度到达测量点时,持续加热一段时间,直至光谱完全稳定,记录该温度下的光谱图。如图13所示,为传感器S-3温度与波长的关系。实验结果表明,在40~80 ℃范围内,样品S-3的温度灵敏度约为15.8/℃,远低于湿度灵敏度。为了更直观地比较温度灵敏度和湿度灵敏度的关系,根据参考文献[21]定义的交叉灵敏度S_C为$S_C=S_T/S_{RH}$,式中S_T为温度灵敏度,S_{RH}为湿度灵敏度。经过计算,传感器的交叉灵敏度为0.108%RH/℃,远低于湿度灵敏度。因此,本实验中,温度对传感器的串扰影响很小。

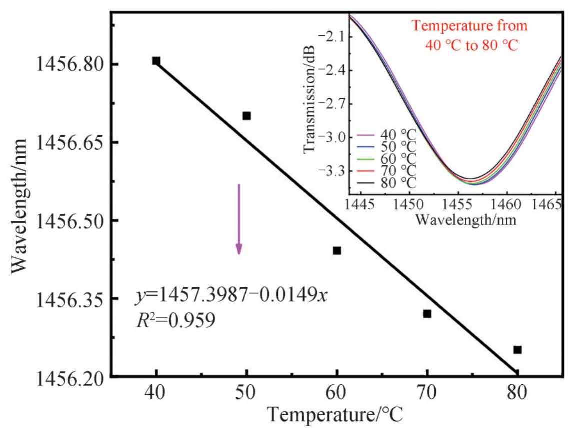


图13　传感器S-1在1 450~1 480处的谐振峰波长随温度变化的光谱图及拟合

Fig.13　Spectral diagram and fitting of resonant peak wavelength variation with temperature of sensor S-1 at 1 450~1 480 nm

4　结论

本文设计制备了一种基于表面涂覆PVA的U型超细光纤湿度传感器,并对未涂覆PVA的U型传感器(S-1)和涂覆PVA的U型传感器(S-2)进行了湿度测试。实验结果发现,由于PVA覆盖层的存在,传感器对周围环境湿度的变化非常敏感。在室温28 ℃下,该传感器在34%RH~90%RH的检测范围内平均湿度灵敏度高达146.1 pm/%RH。该传感器稳定性良好,在湿度增减过程中具有较高的可逆性,温度串扰影响较小,制备方法简单、成本低、便于携带,具有广阔的应用前景。

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U-shaped Microfiber Humidity Sensor Based on PVA Coating

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Abstract: Humidity is one of the important parameters in the field of sensing, and the monitoring and control of humidity is used in many applications, such as agriculture, cultural relics protection, environmental safety, and pharmaceutical engineering. Since traditional electronic humidity sensors are mainly based on capacitance or resistivity measurements, which are more susceptible to electromagnetic interference. Moreover, under high humidity environment, water vapor will corrode the circuit board, which is a great test for the long-term stability of electronic humidity sensors. Compared with traditional humidity sensors, fiber optic humidity sensors have the advantages of corrosion resistance, light weight, can be operated remotely, not subject to electromagnetic interference, etc., and have been widely studied in recent years. Among them, U-shaped microfiber has the advantages of small size, low fabrication cost, high sensitivity, etc., which has a broad prospect in the field of humidity measurement. In this paper, a U-shaped microfiber humidity sensor based on Polyvinyl alcohol (PVA) coating is proposed, and the single-mode optical fiber is pulled into micron-sized microfiber by fiber optic melting and cone pulling machine, and the microfiber with different diameters can be prepared by changing the flame temperature and cone pulling speed, etc. PVA is a strong hydrophilic material, and its refractive index changes with the humidity of the surrounding environment. Due to the high adherence of PVA on the surface of silica, it can be used for the measurement of humidity in a wide range of applications. PVA has high adhesion on the surface of silica, so it can be easily coated on the surface of optical fiber. Because of these special properties of PVA, combining PVA with ultrafine optical fiber can be used to measure humidity. Using the drop coating method, the PVA solution is uniformly coated on the surface of the optical fiber, and the coated ultrafine optical fiber is passed through a homemade mold, which is made of three capillary glass tubes, and the ultrafine optical fiber is fixed into a U-shape by means of UV photoresist and ultraviolet lamp. The paper prepared three humidity sensors, which were used to study the effect of coating thickness on the humidity of the sensors. It was found that the humidity sensitivity of the U-shaped sensors was extremely low when they were not coated with PVA; after coating with PVA, the sensitivity of the sensors increased with the increase of the coating thickness. At the same time, the thicker the coating the greater the loss of the sensor, and it will lead to a longer fabrication time of the sensor, which will cause the PVA droplets on the dropper to evaporate, thus affecting the uniformity of the coating. Therefore, there is a need to select a suitable coating thickness. In order to investigate the effect of the diameter of the waist zone on the humidity of the sensor, two different diameters of the sensor are prepared in this paper. The experimental results found that the smaller the diameter of the waist region, the higher the sensitivity of the sensor, which is due to the fact that the smaller the waist diameter of the ultrafine optical fiber coupled with the external environment the stronger the swift field, which strengthens the interaction between them. The experimental results show that the sensor prepared in this paper has a high humidity sensitivity of 146.1 pm/%RH in the detection range of 34%RH~90%RH. In the humidity measurement experiments, the fluctuation of the temperature will have a certain effect on the experimental results. This is because temperature changes the physicochemical properties of PVA film and U-shaped microfiber, such as changes in refractive index and expansion. Therefore, it is an important work to explore the effect of temperature on the crosstalk of the sensor. Temperature experiments show that the temperature sensitivity of the sensor is 15.8/℃ in the range of 40 ℃~80 ℃, and its crosstalk sensitivity is 0.108%RH/℃, which is much lower than the humidity sensitivity, so the temperature has less effect on this sensor. The sensor designed in this paper has a simple preparation process, high sensitivity, easy to carry, low cost, less influence of temperature crosstalk, which has a wide range of applications in the field of humidity detection.

Key words: Microfiber; U-shaped; Polyvinyl alcohol; Temperature; humidity

OCIS Codes: 060.2370; 060.2430; 060.3510; 120.1880; 130.6010

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