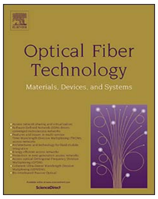
Optical Fiber Technology 41 (2018) 40–47

Contents lists available at ScienceDirect

Optical Fiber Technology

journal homepage: www.elsevier.com/locate/yofte



Optical fiber sensors based on novel polyimide for humidity monitoring of building materials

Jing Chai[a,b,*], Qi Liu[a,*], Jinxuan Liu[a], Dingding Zhang[a,b]

[a] School of Energy and Mining Engineering, Xi'an University of Science and Technology, Xi'an 710054, China
[b] Key Laboratory of Western Mine Exploitation and Hazard Prevention, Ministry of Education, Xi'an 710054, China

ARTICLE INFO

Keywords:
Optical fiber sensor
Relative humidity
Drying
Polyimide coating
Building materials monitoring

ABSTRACT

This paper presents novel preparation methods of polyimide and coupling agent, coated on the fiber Bragg grating (FBG) sensor for monitoring relative humidity (RH). The sensing mechanism that the volume change of the moisture-sensitive polyimide induces the shift of the Bragg wavelength of FBG is used in the RH sensor. The performance of the polymer-coated RH sensor was evaluated under laboratory conditions of temperature over a range of values (20.0–80.0 °C) and humidity over a range of RH values (25.0–95.0%). The time response and RH sensitivity of the sensor based on novel polyimide and coupling agent was improved, compared to the previous. A new packaged RH sensor was designed, which was used in detecting the moisture diffusion and evolutions inside of sample made of building materials which exposed to a controlled environment in the lab after casting. Relative humidity inside of sample with time was 100% in the first phase of vapor-saturated, slowly reduced in the latter phase. The results indicate the RH sensor developed provides a feasible method to detect the influence of environment on moisture inside the material in the drying process.

1. Introduction

Drying, moisture removed from natural or industrial materials down to a specific moisture content, is an important issue in material science and building science as moisture influences several important material properties [1–3]. Moisture migration and self-desiccation are major factors that cause internal RH reduction in materials such as soils and construction materials [4,5]. For predicting the drying time of porous material, good knowledge of the material's moisture diffusion and their evolutions according to different temperature and humidity environments is a necessity. The measurement of humidity and moisture is significant for a broad range of practical applications including civil engineering, soil humidity monitoring, food processing industry, medicine, mineral processing and so on [6,7]. There are several conventional solutions to measure relative humidity, such as hygrometric, capacitive and resistive sensors. The limitation of conventional electric sensors involves bad resistance to corrosion, high cost, and incapacity to be used in inflammable or explosive environments for high sensitivity to electromagnetic interference. On the contrary, the optical fiber sensors, showing particular characteristics, can overcome these limitations. It is convenient for multiplexing a great number of different kinds of sensors and constructing sensing network, reducing the multiple cabling employed in conventional electrical sensors, especially in harsh environments where conventional electrical sensors are supposed to be inadequate or would not operate [8–10].

The different kinds of RH sensors based on optical fiber sensing techniques, which can be categorized according to the schemes on which they rely, include direct spectroscopy [11–13], in-fiber grating [14,15], absorption techniques [16–18], interferometric techniques [19–21] and evanescent wave methods [22–24]. Yeo et al. [25]investigated the characteristics of polymer-coated FBG humidity sensor, which was including RH sensitivity, temperature sensitivity, and the time response. Berruti et al. [26]reported polymer-coated FBG-based sensors, exposure to ionizing radiation up to 10 kGy, were able to measure relative humidity, shown their capability as an effective selective to replace electronic sensors in high energy applications. Swanson et al. [27] studied that the moisture response of polymer-coated FBG-based humidity sensor could be improved by adding more water associating sites and reducing carbon chain lengths to modify the chemical structure of polyimide. The foundation of these FBG-based relative humidity sensors is the use of an appropriate chemical material to make a mechanical effect on the FBG. The volume change of material, which is coated directly on the FBG, has a significant influence on the shift of the Bragg wavelength. The polyimide has been investigated as a suitable relative humidity responsive coating with a great sensitivity to moisture. However, there are still a few issues to overcome,

---

[*] Corresponding authors at: School of Energy and Mining Engineering, Xi'an University of Science and Technology, Xi'an 710054, China.
E-mail addresses: Chaij@xust.edu.cn (J. Chai), Lqxust@163.com (Q. Liu).

https://doi.org/10.1016/j.yofte.2017.12.013
Received 13 June 2017; Received in revised form 6 December 2017; Accepted 17 December 2017

about increasing solubility, enhancing interfacial adhesion between polymer and FBG, and improving the preparation efficiency.

The aim of this work is to investigate an innovative preparation of polyimide, the surface characteristic of the polyimide coating, and the sensing performance of polymer-coated FBG-based RH sensors, which can be used to determine the moisture diffusion and their evolutions of the sample made of building materials exposed to environmental conditions in the drying process.

2. Sensing principle

The structure of FBG can be manufactured when a modulation of the refractive index is made within the fiber core, which has been discussed by different investigators [28]. When a broad band pulse is spread down the FBG which serves as a wavelength selective filter, the most is transmitted and a light signal with a specific wavelength is reflected, called the Bragg wavelength (λ_B). The Bragg reflection wavelength is strictly dependent on the interval of the grating plane in the fiber (Λ) and the fiber effective refractive index (n_{eff}), as given by Eq. (1).

$$\lambda_B = 2n_{eff}\Lambda \tag{1}$$

The FBG-based sensor is adaptive for temperature and strain measurements. In most cases, compared with the spacial period of the grating, the change of the effective refractive index serves as a decisive effect on the FBG. Due to the thermo-optic effect and the thermal expansion, the change of temperature undergone by the FBG has a significant effect on the effective refractive index and the grating spatial period, leading to a shift in the Bragg wavelength. The axial strain has the same effect on account of the elasto-optical effect and the elastic behaviour. The variation of temperature and strain experienced by the FBG has an impact on the refractive index and the spacial period of the grating, leading to a change in Bragg wavelength, which can be expressed as

$$\frac{\Delta\lambda_B}{\lambda_B}=(1-P_e)\varepsilon + [(1-P_e)\alpha + \zeta]\Delta T \tag{2}$$

where P_e represents the photoelastic constant of the fiber, ε the axial strain, ζ the thermo-optic coefficient of the fiber, α the fiber thermal-expansion coefficient, and ΔT the change of temperature.

Based on the basic principle of the FBG, the relative humidity sensor exploits the strain sensing characteristic by coating the FBG with a water sensitive polymer, expanding with moisture immersion, to measure the change in humidity. The volume change of the moisture sensitive layer imposes the direct strain effect on the fiber, and the shift of the Bragg wavelength is determined by relative humidity and temperature, which can be expressed by

$$\frac{\Delta\lambda_B}{\lambda_B}=(1-P_e)C_{RH}\Delta RH + (1-P_e)C_T\Delta T + \zeta\Delta T \tag{3}$$

$$C_{RH} = \left[1-\frac{E_F r_F^2 C_0}{E_F r_F^2 + E_P(t^2 + 2tr_F)}\right]M_P \tag{4}$$

$$C_T = \frac{E_P(r_F + t)^2}{E_P(r_F + t)^2 + E_F r_F^2}(W_P-W_F) \tag{5}$$

where C_{RH} , C_T represent the relative humidity and temperature sensitivity, respectively, W_P, W_F are the thermal expansion coefficients of the polymer and fiber, respectively, M_P is the moisture expansion coefficient of the polymer, E_P, E_F are the Young's modulus of the polymer and fiber, r_F is the radius of fiber, t is the thickness of the polymer, and C_0 is the interface coherent coefficient between the fiber and the polymer.

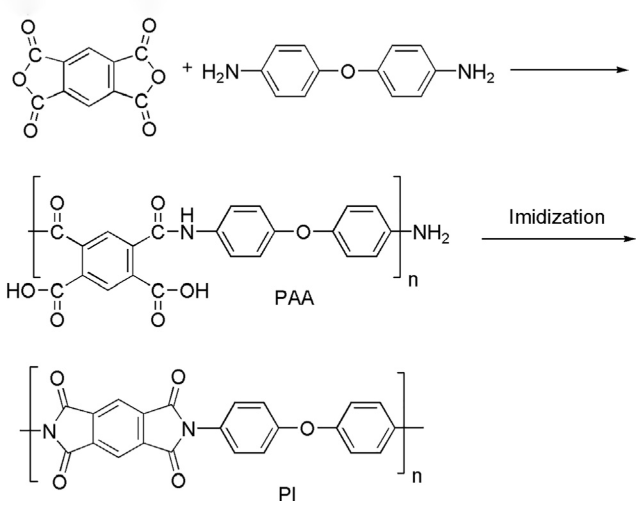


Fig. 1. Schematic illustration of the preparation of polyimide.

3. Sensor fabrication, packaging and calibration

3.1. Materials and methods

A certain volume of N-methylpyrrolidone (NMP) was added to a 250 mL three-necked flask at room temperature, while nitrogen was inlet, a quantity of 4,4′-diaminodiphenyl ether (ODA) was added and stirred to dissolve completely. According to the metering ratio, a slightly excessive benzenetetracarboxylic anhydride (PMDA) was added to the three-necked flask in batches, (the molar ratio of PMDA to ODA is 1.02: 1). The mixture was stirred at a constant rate for 6 h to obtain a transparent and flaxen polyamide acid (PAA) solution. Subsequently, the chemically imidization was conducted by a certain amount of a mixed solution of acetic anhydride and pyridine. After stirring for 24 h, the pale yellow polyimide (PI) solution was obtained. During the PI solution configuration, the chemical reaction process is shown in Fig. 1.

The pretreatment of fiber Bragg gratings is helpful to improve the bonding ability between the polyimide film and the Bragg grating region. For enhancing the stress transfer of the polyimide to the attached grating, N-hydroxyethyl ethylenediamine was chosen to be coupling agent, which could be combined with the hydroxyl group of the fiber Bragg grating cladding and the amino group of the polyimide. The chemical expression of N-hydroxyethyl ethylenediamine is $C_4H_{12}N_2O$, which is buff, transparent and viscous liquid. N-hydroxyethyl ethylenediamine has hygroscopicity, strong alkaline, slightly ammonia smell, and it can be miscible with water and alcohol, slightly soluble in ether. N-hydroxyethyl ethylenediamine and deionized water were mixed at a concentration of about 0.05%. First, the surface of fiber Bragg grating was scrubbed to ensure that the fiber surface is clean, then the clean fiber Bragg grating was immersed into the N-hydroxyethyl ethylenediamine solution for 3 min, and then the residual N-hydroxyethyl ethylenediamine solution of the surface was dried. The principle of strengthening the fiber grating gate region and polyimide coupling with N-hydroxyethyl ethylenediamine is shown in Fig. 2.

The PI solution was coated on the bare FBG which has a gate length of 30 mm, and the coated FBG was heated to 200 °C to form a thin film. The specific process is as follows: The coating speed is controlled at 0.1 mm/s. Firstly, put the FBG which plated with PI film into the electric blast oven to heat, during the warming period, the heating rate is 1 °C/min from room temperature to 80 °C. In order to ensure inside

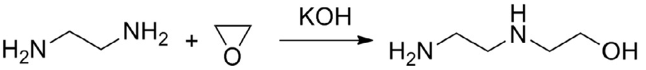


Fig. 2. Illustration of the synthesis of N-hydroxyethyl ethylenediamine.