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SiO_2-doping Films Effects on Optical Properties of Optical Fiber Surface Plasmon Resonance Hydrogen Sensor

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Abstract. SiO_2-doping WO_3 films were obtained by magnetron sputtering. Experiments indicated more obvious optical phenomenon with SiO_2-doping films. Different thickness of the SiO_2 film was discussed for better optical properties, according to the resonance peak width, the resonance depth and the sensitivity. This work can provide theory support for the design of optical fiber surface plasmon resonance hydrogen sensor.

1. Introduction

In recently years, hydrogen energy has attracted a great deal of attentions as a renewable and abundant potential clean energy source for the next generation of auto mobiles and household appliance because of its clean combustion without any release of pollutants[1-2]. But hydrogen is flammable and explosive, the lower explosion limit of hydrogen is 4% in air[3]. Therefore, the preparation of hydrogen sensors used for online and real-time detection is especially important. Development of hydrogen senors with high sensitivity, stability, and selectivity are highly desired[4-5]. Optical fiber hydrogen sensor is an ideal candidate which can measure hydrogen concentration by optical methods. The surface plasmon resonance(SPR) technology has the advantages of high safety, reliability, sensitivity for hydrogen measurement compared with conventional gas detection technique[6]. In order to obtain high sensitivity, it is very essential to research hydrogen sensitive films. Tungsten trioxide(WO_3) is an important material for optical fiber hydrogen sensor. WO_3 thin films will change the color from greenish yellow to blue when exposed to hydrogen. Then hydrogen can be detected by monitoring the changes in optical transmittance. Many researches show Pd or Pt is a good material, which can improve hydrogen selectivity to WO_3 [7-8].

The addition of the adjustment layer SiO_2 can make the SPR phenomenon more obvious, which is very important for the optical fiber SPR hydrogen sensor. In this paper, we got WO_3 thin films and SiO_2-doped WO_3 thin films using magnetic sputtering process. Hydrogen-sensing properties of the thin films had been investigated during alternate exposures to different gas concentrations of H_2-N_2 mixture and air at room temperature. The experiment of optical properties demonstrated that SiO_2-doped WO_3 thin films prefers significantly better than WO_3 thin films.

2. Experiment

Composite thin films can be prepared by several methods, including magnetron sputtering, electron beam, sol-gel, etc[6]. Compared with other methods, the quality of the films obtained by magnetron sputtering is better. In the experiments of fabricating hydrogen sensitive films for the optical fiber SPR sensor, WO_3 was used as the basic materials, and the SiO_2-doped WO_3/Pt films were prepared by magnetic sputtering method.

Firstly, the surface cladding of the optical fiber was removed by chemical reaction with hydrofluoric acid. Then thin films were coated layer by layer using magnetron sputtering technique, as shown in the Figure 1. Ag and Pt films are used as metal layer and catalytic layer, respectively.

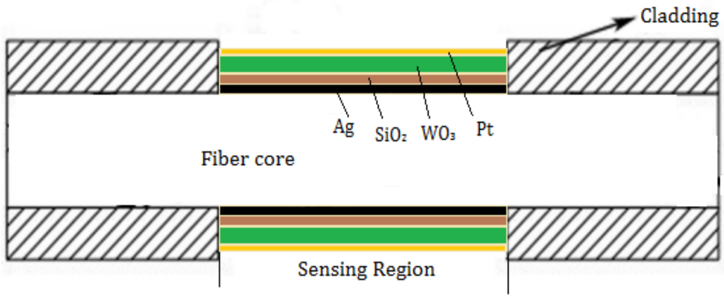

Figure 1. Multiple Films Mode

In the sputtering process, the corresponding process parameters need to be set for each layer. Table 1 is the process parameters for depositing SiO_2 film.

Table 1. Process parameters for depositing SiO_2 film

Sputtering Target	SiO_2
Starting Power	100 W
Sputtering Power	267 W
Working Gas	Ar
Working Pressure	0.005 mbar

The morphology of the samples were observed using electron microscope. Figure 2 is the films surface of 35nm Ag, 100nm SiO_2, 180nm WO_3 and 3nm Pt , respectively. Because the Pt film is too thin, it can't be seen. Other films can be observed clearly.

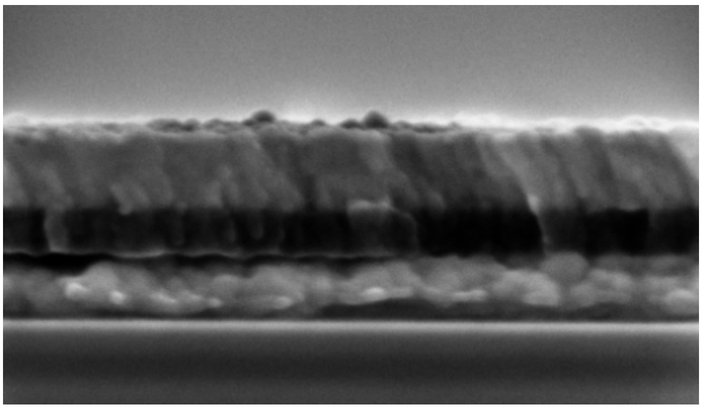

Figure 2. Fracture surface morphology of the films

The hydrogen sensitive properties of the films were tested. The results show that the optical properties with the SiO_2-doped WO_3 films are improved compared with the WO_3 films without SiO_2, and the sensitivity of the films is satisfactory in the hydrogen concentration range of 0 ppm to 40000 ppm.

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3. Results and discuss

At the same context, the SiO_2-doped thin film exhibited bigger sensing effect. With the same concentration of 4% H_2 gas, the more pronounced improve effect in sensitivity is shown Figure 3. The green curve is the SPR curve with the films, including Ag, WO_3 and Pt. The blue curve is the SPR curve by adding SiO_2 film.

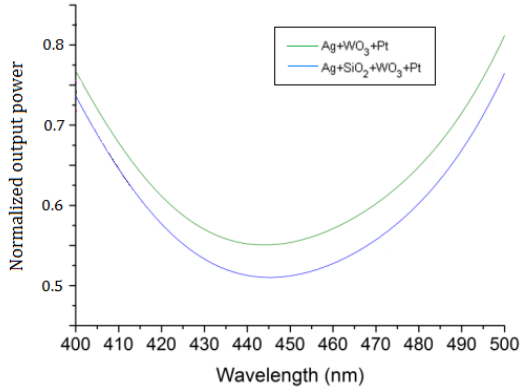

Figure 3. SPR curves with different sensing layers

It is also found that the resonance depth of the sensor increased with the increase of the thickness of the SiO_2 film, and the resonance wavelengths drifted toward longer wavelengths, as shown in the Figure 4. However, when the thickness of SiO_2 film is greater than 100nm, the resonance depth decreases slightly with the increase of SiO_2 film thickness. For this reason, several parameters affected the optical fiber SPR hydrogen sensor are considered.

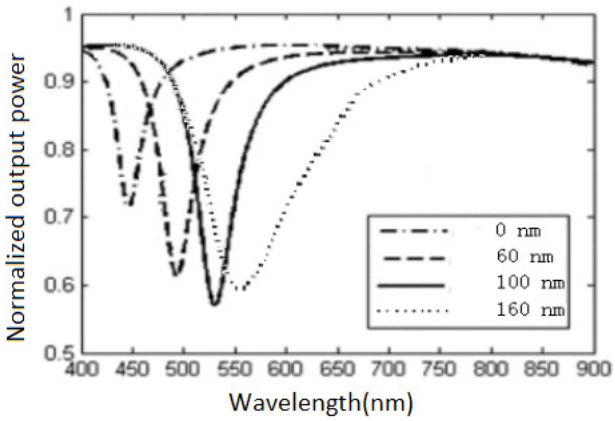

Figure 4. The SPR curve with different thickness SiO_2 film in 4% hydrogen condition

The properties of optical fiber SPR hydrogen sensor based on wavelength modulation can be described by three basic characteristic parameters, namely, the resonance wavelength, the resonance peak width and the resonance depth. In this paper, the resonance peak width is defined as the spectral width at half the sum of the maximum and minimum of the resonance peak, which is simply RS. The depth of resonance is defined as the difference between the peak value and the corresponding peak value of the two peaks and valleys, which is roughly written as RD. Sensitivity is defined as the drift of the resonance wavelength caused with the change of hydrogen concentration, which is simply S. In order to directly compare the overall optical properties of the optical fiber SPR hydrogen sensor, a comprehensive factor is also introduced, that is TF.

$$TF = (S / RW) * RD \quad (1)$$

In the formula, S/RW represents the signal to noise ratio of the sensor. The composite factor is the product of the signal to noise ratio and resonance depth. The thickness of SiO_2 layer is designed according to the composite factor.

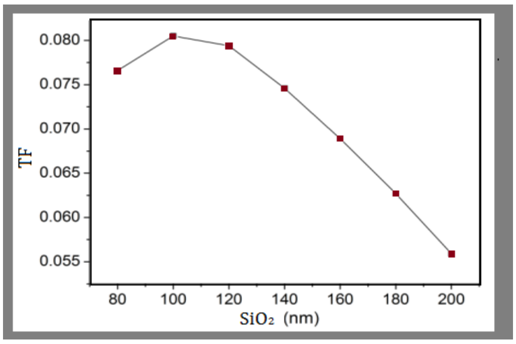

Figure 5. The TF variation curve with the thickness of SiO_2

As shown in Figure 5, with the increase of the thickness of the SiO_2 film, the composite factor of the sensor increases first and then decreases, and reaches the maximum at about 100 nm, which is most suitable for the optical fiber SPR hydrogen sensor.

4.Conclusion

SiO_2-doped WO_3 optical hydrogen-sensing thin films were prepared by magnetron sputtering. The optical properties of the sensitive films for optical fiber SPR hydrogen sensor were studied in this work, and it was found that the optical properties were more obvious with SiO_2-doped thin films, so the hydrogen concentration can be easily detected.

Acknowledgments

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