

HUMIDITY SENSING BY LOVE WAVE DETECTORS COATED WITH DIFFERENT POLYMERIC FILMS

Li-jun Wang, Jian-sheng Liu*, Shi-tang He

Institute of Acoustics, Chinese Academy of Science, Beijing 100190, China

*Corresponding author, E-mail: liujiansheng@mail.ioa.ac.cn; Tel.: 86-010-82547803.

In this paper, an experimental investigation was performed on humidity sensing by using Love wave devices coated with different polymeric layers. The Love wave device consists of two interdigital transducers (IDTs) with a period of 28 μm , an ST-90°X quartz substrate. Three different films of SU-8, SU-8+PVA and PVA, were spin-coated on the substrate surface by a sol-gel process as the waveguide layer and the sensing film. A detailed description was presented for the preparation of the solution and coating and curing process of SU-8 and PVA. A network analyzer was used to measure the operation frequency and insertion loss of the sensor at different relative humidity. The absorption mechanism of polymeric layers was analyzed and the mechanism was successfully used to explain the experimental results of the humidity sensing of Love wave devices coated with SU-8, SU-8+PVA, or PVA layers.

Keywords: Surface acoustic wave; Love wave; Humidity sensor; PVA; SU-8; Relative humidity

1. INTRODUCTION

Surface acoustic wave (SAW) humidity sensor[1-2] has many advantages, such as high sensitivity, wide detection band, and high frequency, so it is increasingly widely used in food storage and processing, weather forecast, medicine, etc.

According to the type of acoustic excitation, SAW humidity sensor includes Rayleigh wave type and Love wave type sensor. Compared with Rayleigh wave sensor, which is the current commonly used, Love wave humidity sensor has the following characteristics: Firstly, Love wave sensor can be used in the liquid phase detection[3] because of that Love wave has only horizontal shear direction of particle displacement and the loss is low while working in liquid medium. Secondly, Love wave sensor has higher sensibility[4] for there is a layer of waveguide above the substrate and IDT which can accumulate the energy at the waveguide and the substrate surface as well as reduce the corrosion of metal IDT.

Polymer materials are common used as acoustic waveguide layer. Polymer waveguide (such as SU-8) has high sensitivity, the shear velocity is low, and coating process is relatively simple. Polyvinyl alcohol (PVA) is a kind of commonly used polymer hygroscopic material^[5], which has good hygroscopicity and is generally made by the spin coating method.

Based on Love wave device which was fabricated on a ST-90°X quartz substrate on whose surface respectively coated with a layer of the SU-8, SU-8+PVA,

PVA three different membranes as a waveguide and moisture sensitive layer, this paper described experimental research on the relative humidity in the space. The Love wave humidity sensor experiment device was set up and the frequency offset and loss offset was measured by the network analyzer under different relative humidity. Finally, this paper analyzed the results of the Love wave device which coated three different membranes, and compared the differences between them.

2. EXPERIMENTAL METHOD

2.1. Fabrication of Love wave devices

The structure of Love wave device this paper used is shown in figure 1. A piezoelectric ST-90°X quartz substrate with a pair of Al-film Interdigital transducers (IDTs) and the films deposited on the substrate surface formed the Love wave delay line. The device parameters of the IDT used in this article are as follows: acoustic aperture is 2 mm; Center distance is 4 mm; IDT cycle is 28 μm , each IDT has 72 cycles; the thickness of the electrode is 200 nm. Love wave device is fabricated on a ST-90°X quartz substrate and the operating frequency is about 178 MHz.

A layer or two layers of film (SU-8, SU-8+PVA, PVA) are coated onto the substrate. Wherein the layers act as the guiding layer and the sensing film, which was spin-coated on the substrate surface by a sol-gel process.

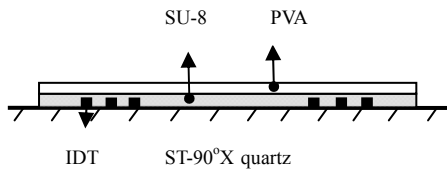


Fig.1. Delay line structure of Love wave humidity sensor

PVA has a lot of strong hydrophilic hydroxyl (-OH) which can form hydrogen bonds with water molecules. The commercial PVA used in the experiment (flocculent, average degree of polymerization is 1750 ± 50) was provided by Beijing Yili Fine Chemicals Company. Specific steps of the preparation of PVA solution are as follows: Firstly, dilute 30 g of PVA floccules in 600 ml of deionized water contained in a glass beaker heated at 60 °C for 40 min^[6-7] and shaken by a magnetic stirrer during the treatment time. Secondly, adjust the temperature to mount into 90 °C slowly and stir by a magnetic stirrer for about 50 minutes. The heating process was stopped when the solution became dense and no longer contained small particles and the solution was left to cool in air for at least 3 h. This was spin-coated onto Love wave device by a sol-gel process using drops (5 ml) of the prepared PVA solution by spinning at 2000 rpm for 50 s with a post-curing of PVA film at 60 °C in air for 30 min. The PVA film totally covered the Love wave device. The thickness of the prepared film was about 0.72 μm , which is about 2.57% of the acoustic wavelength.

SU-8 solution was prepared by mixing the SU-8 2050 type of photoresist and special diluents (produced by Microchen Company) according to the ratio of 1:3. This was spin-coated onto Love wave device using drops (5 ml) of the prepared SU-8 solution by spinning at 2000 rpm for 50 s with a post-curing of SU-8 film at 150 °C for 30 min. The SU-8 film totally covered the Love wave device. The thickness of the prepared film was about 0.74 μm , which is about 2.64% of the acoustic wavelength. The film thickness can be controlled by the rotation rate and spinning time.

2.2. Set up the experimental platform

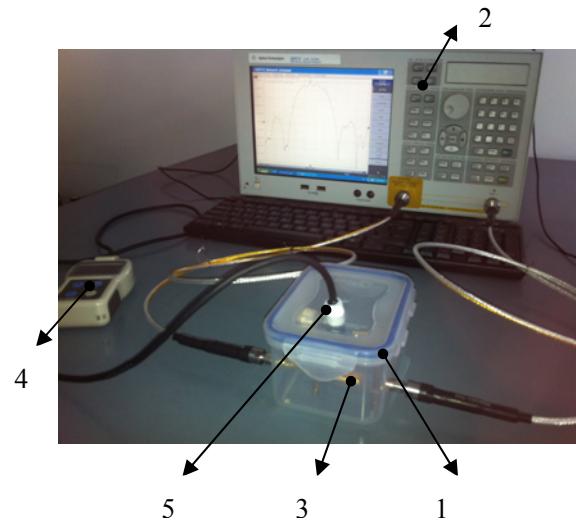


Fig.2. Experimental facility ((1) Seal box, (2) network analyzer, (3) Love wave device, (4) hygrometer, (5) hygrometer probe)

The experimental platform set up in this paper is shown in figure 2. Love wave device welded on a small piece of matching circuit boards was placed into a closed seal box. The air humidity in the box varied slowly by inserting a small piece of wet sponge (for humidity increase) or hygroscopic silica gel (for humidity decrease). The digital thermo-hygrometer (OMEGA RH32B-C2) monitored variations in relative humidity (RH). The network analyzer (Agilent E5071C) measured frequency offset and loss offset of the Love wave device under different relative humidity environment in the seal box. The measurements were performed at the room temperature.

3. RESULTS AND DISCUSSION

3.1. Results

The results are shown in Figure 3 and Figure 4, where “*” showed the results of SU-8 film-device; “ ∇ ” showed the results of SU-8 + PVA films-device; “o” showed the results of PVA film-device.

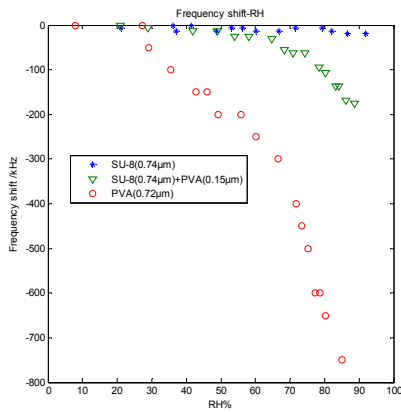


Fig.3. Frequency offset - Relative humidity

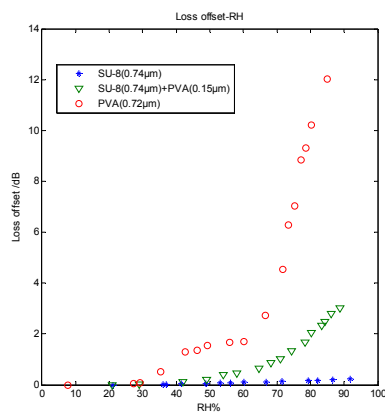


Fig.4. Loss offset - Relative humidity

Figure 3 shows the relationship between frequency offset and relative humidity and Figure 4 shows the relationship between loss offset and relative humidity. For the SU-8 film-device: The thickness of SU-8 film was $0.74 \mu\text{m}$. The Figure 3 and 4 shows that there is no obvious relationship between relative humidity and frequency of Love wave device which coated SU-8 film. It can be concluded that: the frequency of Love wave device which coated SU-8 film has no obvious reaction to humidity in the space. In addition, the minimum loss of SU-8 film-device is about 12 dB. For the SU-8 + PVA films-device: The thickness of SU-8 film was $0.74 \mu\text{m}$ and PVA film was about $0.15 \mu\text{m}$. The frequency of device coated $0.74 \mu\text{m}$ thickness of SU-8 film and about $0.15 \mu\text{m}$ thickness of PVA membrane has obvious reaction to the change of relative humidity and the loss offset is apparent. Frequency shift versus relative humidity: At low relative humidity ($\text{RH} < 48\%$), the frequency offset of device is small (frequency shift $< 12 \text{ kHz}$). For $48\% < \text{RH} < 70\%$, the frequency offset increases gradually. At high relative humidity ($70\% < \text{RH} < 89\%$), the frequency offset increases rapidly and

the increasing rate of frequency offset at $\text{RH} = 85\%$ is $15 \text{ kHz/RH}\%$. The maximum frequency offset at $\text{RH} = 89\%$ is 175 kHz . Loss shift versus relative humidity: At low relative humidity ($\text{RH} < 48\%$), the loss offset of device is small (loss offset $< 0.2 \text{ dB}$). For $48\% < \text{RH} < 70\%$, the loss offset increases gradually. At high relative humidity ($70\% < \text{RH} < 89\%$), the loss offset increases rapidly and the maximum loss offset at $\text{RH} = 89\%$ is 3 dB . For the PVA film-device: The thickness of PVA film was about $0.72 \mu\text{m}$, which is about 2.57% of the acoustic wavelength. The frequency of device coated $0.72 \mu\text{m}$ thickness of PVA membrane has more obvious reaction to the change of relative humidity and the loss offset is more apparent than that of device coated SU-8+PVA double membranes. Frequency shift versus relative humidity: At low relative humidity ($\text{RH} < 30\%$), the frequency offset of device is small (frequency shift $< 50 \text{ kHz}$). For $30\% < \text{RH} < 60\%$, the frequency offset increases gradually. At high relative humidity ($60\% < \text{RH} < 88\%$), the frequency offset increases rapidly and the increasing rate of frequency offset at $\text{RH} = 76\%$ is $50 \text{ kHz/RH}\%$. The maximum frequency offset at $\text{RH} = 89\%$ is 750 kHz . Loss shift versus relative humidity: At low relative humidity ($\text{RH} < 30\%$), the loss offset of device is small (loss offset $< 1 \text{ dB}$). For $30\% < \text{RH} < 66\%$, the loss offset increases gradually, but the biggest shift is still lower than 3 dB . At high relative humidity ($66\% < \text{RH} < 88\%$), the loss offset increases rapidly and the maximum loss offset at $\text{RH} = 88\%$ is 12 dB .

It can be concluded that: Firstly, the frequency offset of the device coated SU-8+PVA films is better than SU-8 device. But the overall frequency offset is small. In addition, the loss of SU-8+PVA double membranes device increased to 13.5 dB and increased by 1.5 dB than the loss of SU-8 film device; Secondly, the frequency offset of Love wave of humidity sensor device coated PVA monolayer film is large and the maximum frequency offset is 750 kHz which reached the ideal value. The maximum loss offset 12 dB is also within the scope of permissible. In addition, the loss of Love wave humidity sensor device of PVA monolayer membrane is small, about 6.5 dB .

3.2. Discussion the hygroscopicity of SU-8 and PVA

From the experimental results, the SU-8 film has almost no hygroscopicity but PVA membrane has good hygroscopicity among various waveguide layers and sensing films of Love wave humidity sensor.

To search papers to determine whether SU-8 structure containing groups of moisture absorption. The structure of SU-8 is shown in figure 5(a). It is shown

from the molecular structure of SU-8, molecular formula of SU-8 monomer structure contains no hygroscopic groups. The structural formula of PVA is shown in figure 5 (b). PVA is a very versatile water-soluble polymer and has a lot of strong hydrophilic hydroxyl (-OH). Hydroxyl groups of PVA can form hydrogen bonds with water molecules therefore PVA is a kind of commonly used polymer hygroscopic material. Hygroscopicity of PVA is reversible and it is a kind of physical adsorption.

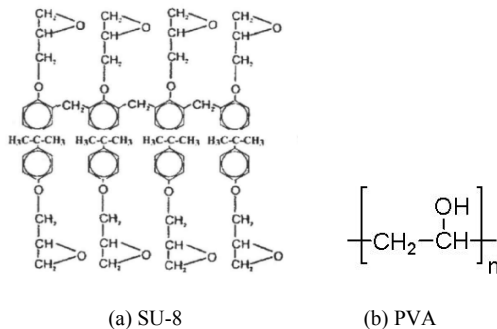


Fig.5. Molecular structure of SU-8 and PVA

From the above analysis, Love wave sensor coated SU-8 film has no obvious response to humidity because the SU-8 film contains no hygroscopic groups; the device coated PVA monolayer membrane on the response of the relative humidity is better. In addition, the loss of Love wave sensor coated SU-8 monolayer film is big; the loss of device coated SU-8+PVA double membranes increases further, the device coated SU-8+PVA double membranes can be viewed SU-8 as the waveguide layer while PVA as moisture absorption layer, SU-8 is polymer waveguides, the loss will be increased with the increase of the waveguide layer thickness, so choosing the SU-8 as the waveguide layer of Love wave humidity sensor is not ideal. However, the loss of device coated PVA monolayer membrane is about 6.5 dB and smaller than that of SU-8+PVA double membranes device.

Common rules exist in the response to the humidity of SU-8+PVA double membranes device and PVA monolayer membrane device: At low relative humidity, the frequency offset and loss offset of device are small. For about $40\% < RH < 60\%$, the frequency offset and loss offset increase gradually. At high relative humidity, the frequency offset and loss offset increase rapidly. This is because there are a lot of hydroxy groups in the polyvinyl alcohol which can form hydrogen bond with water. The reaction belongs to the physical adsorption and can be multi-layer adsorption of water molecules. However, it can form strong hydrogen bonds between water molecules so once the PVA membrane has

absorbed of water molecules in the surface, the second and the third layer of adsorption are very easy to form.

4. CONCLUSIONS

In this paper, an experimental investigation was performed on humidity sensing by using Love wave devices coated with different polymeric layers. SU-8, SU-8+PVA, PVA three different films were spin-coated on the substrate surface as the waveguide layer and the sensing film. A network analyzer was used to measure the operation frequency and insertion loss of the sensor at different relative humidity. The absorption mechanism of polymeric layers was analyzed and the mechanism was successfully used to explain the experimental results of the humidity sensing of Love wave devices coated with SU-8, SU-8+PVA, or PVA layers.

ACKNOWLEDGEMENTS

This research is supported by National Science Foundation of China (No. 11104314).

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