Thin polymer film based rapid surface acoustic wave humidity sensors

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ABSTRACT

Polyvinyl alcohol (PVA) and polyvinyl pyrrolidone (PVP) thin films, deposited on the surface of glass slides, were studied using transmission FTIR spectroscopy upon varying relative humidity (RH) from 2 to 70%. The obtained data revealed fast dynamics of water vapor adsorption–desorption with responses on the order of several seconds. Based on the fast FTIR signal intensity changes versus RH, it was proposed that a similar rapid response can be achieved for PVA and PVP coated SAW devices due to changes in mass-loading and film viscoelastic properties upon absorption of water vapor in the films. Sub-micron thickness films were spin-coated onto the surface of $LiNbO_3$ SAW substrates. Both PVA and PVP based humidity sensors revealed prompt reversible response to variations in humidity, although PVP-based device demonstrated better sensor parameters with total insertion loss variation of about 50 dB over the studied RH range and response time 1.5 s for the humidity step 5–95% (recovery time – 2.5 s), representing one of the fastest SAW-based humidity sensors reported to date.

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1. Introduction

The development of SAW humidity sensors has attracted much attention in recent years [1] due to combination of such properties as high sensitivity to humidity [2] and possibility of easily realizable wireless control via antenna installed on the sensor device [3]. In this way, the sensor itself can also be passive, i.e. electrical power supply is only needed on the interrogator, while the sensor chip is powered only by the radio frequency (RF) signal of the interrogator. One of the key components of the SAW humidity sensor is the sensing coating, which should be able to reversibly absorb water molecules and exhibit a change in film properties that produces a measurable sensor response. Various polymeric materials are commonly used to make sensitive coatings for SAW humidity sensors, among them poly(N-vinylpyrrolidone) (PVP) [4], poly(vinylalcohol) (PVA) [5,6], conjugated organic and organometallic poly-ynes [7,8], polyXIO [9] and others. Although polymer based humidity sensors have been developed, the response/recovery time for many polymer coatings were shown to be rather long, frequently on the order of several seconds or even minutes [5,6,10]. At the same time, for such applications as medical diagnostics [11–13] and meteorology [14] it is necessary to be able to conduct humidity measurements and data acquisition

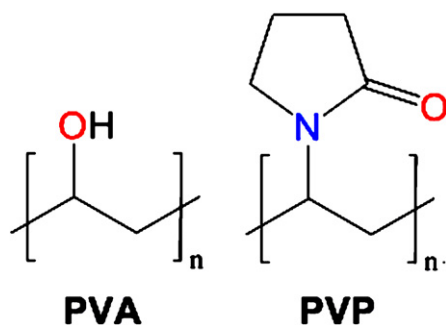in real time, hence, prompt sensor response dynamics becomes an essential parameter [11].

In this paper, we report rapidly responding SAW sensors based on thin PVA and PVP films (Scheme 1). Specially designed $LiNbO_3$-based SAW delay line substrates patterned with aluminum transducers and a reflector (Fig. 1) were designed and tested in our lab. A simulation of the SAW response for our sensor devices (without polymer coating) was performed according to standard coupling-of-modes (COM) and cascaded P-matrix theory. The theoretical treatments of Martin, Grate and others found in the literature [15,16] were considered when evaluating the potential mass sensitivity and viscoelastic behavior of the polymer coated sensors.

Evaluation of the water adsorption–desorption behavior of the polymer films under varying humidity conditions was studied using transmission FTIR, which revealed effective reversible water loading of the studied polymers upon successively increasing and decreasing RH%. It was shown that FTIR measurements can be used for approximate assertion of the response dynamics in the case of thin polymer films.

Study of SAW sensor operation revealed rapid and sensitive sensors in the case of both PVA and PVP coated devices, however, PVP-based humidity sensors generally demonstrated better performance, with insertion loss varying by about 50 dB over the RH range 20–100%, and response and recovery times for RH step from 5 to 95% being 1.5 s and 2.5 s, respectively. To the best of our knowledge, the obtained PVP-based SAW humidity sensor appears to be among the fastest SAW humidity detectors, reported to date.

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Scheme 1. Structure of PVA and PVP polymers used for fabrication of humidity sensitive layers.

2. Experimental

2.1. Materials

Both polymer powders were purchased from Sigma–Aldrich and used as received. PVA powder is 80% hydrolyzed, with a molecular weight of 9000–10,000 Da. The PVP powder was reported to have a molecular weight of 1,300,000 Da. Solutions of the polymers were prepared by dissolving 0.4 g of PVA in 40 ml of deionized water (Ultrapure water system, Barnstead) and 0.45 g of PVP in 15 ml of ethanol (Pharmco-AAPER), respectively.

2.2. Preparation of samples for FTIR evaluation

Square-shaped glass substrates (1 × 1 inch) were cut from pre-cleaned glass microslides purchased from VWR International. All glassware and glass substrates were cleaned by piranha solution (3:1 (v/v) concentrated sulfuric acid/30% hydrogen peroxide) (*Caution: piranha solution is extremely corrosive and can react violently with organic compounds; gloves, goggles, and face shields should be used for protection.*) and dried by a stream of nitrogen before use. Deposition of the polymer films was carried out using EC101D Photo-resist Spinner Controller (Headway Research Inc.) at rates of 7000 and 10,000 rpm, for 30 s, using a drop volume of 30 µl.
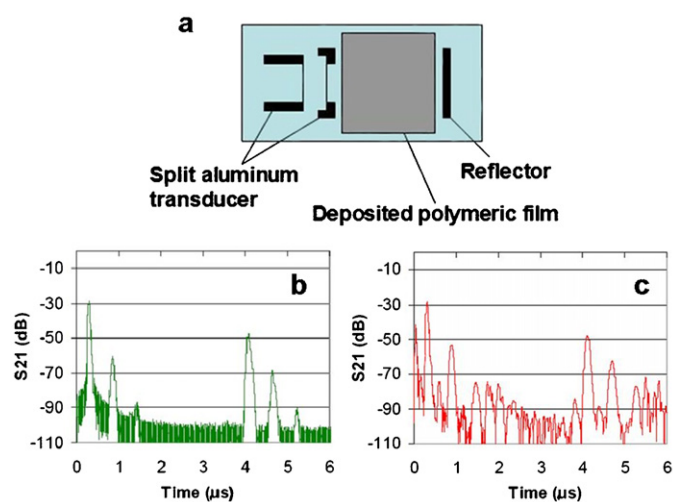

Fig. 1. Schematic of the SAW device designed in this work (a); simulation of the SAW response for uncovered device (b) and experimental SAW response for uncovered device (c).

2.3. Preparation of humidity sensors

The SAW device fabricated for our tests consists of two closely spaced aluminum split-electrode transducers near one end of the die, each 12λ long, with a 15λ non-split electrode reflector located at the opposite end of the die (Fig. 1). The acoustic aperture is 100λ, and the delay line is 460λ. The center frequency of the device was selected as 250 MHz for convenience and YZ cut $LiNbO_3$ (Crystal Technology, Inc.) was used as a substrate. All the SAW substrates were O_2 plasma treated (100 mW RF power, 400 mTorr of O_2, 30 s). Spin-coating deposition of the films was performed according to the procedure described above for the FTIR samples. In order to prevent deposition of polymeric material onto electrodes, they were protected using adhesive tape which was removed after spin-coating.

2.4. Measurements

Thickness measurements of the films were performed on the final SAW devices using PicoPlus AFM (Molecular Imaging, Temple, AZ) and a contact mode silicon nitride AFM tip (NP-S20, force constant 0.12 N/m, Veeco). All the AFM experiments were done in ambient atmosphere at room temperature. In this procedure, a razor blade was used to make a clean cut through the film and down to the hard $LiNbO_3$ substrate. The obtained film edge was located using an optical microscope then scanned with the AFM tip to produce a profile of the step. Thickness was determined using the AFM cross-section. In order to obtain statistical results, several cuts were made in different areas of the film and then each cut was AFM-imaged in several different spots. The obtained thickness values were averaged and the standard deviation was found.

The transmission FTIR studies of water vapor adsorption–desorption behavior of the film deposited on glass substrates were performed on a Tensor 27 (Bruker) spectrometer. Data collection and processing was performed using Opus Software (Bruker Optics). IR spectra were collected every 3 s while simultaneously changing the ambient humidity conditions. Glass slides with deposited films were placed in a special constructed hermetically sealed chamber with two CaF_2 windows allowing the IR beam passing through the sample inside it. The chamber was connected to a gas line which allowed control of the humidity level inside the working space. The humidity level inside the chamber was measured using a commercial humidity-temperature recorder (Omega CTH89).

Measurements of the fabricated SAW sensors were carried out utilizing an RF probing station (Wentworth Laboratories MP-0900) connected to a network analyzer (Agilent E5070B). Humidity was controlled by adjusting the mixed ratio of dry to wet nitrogen flows, using a line similar to that above described for IR tests. Control of the humidity level and temperature in the gas line was performed using an Omega RH32B commercial analyzer. The network analyzer plot of the device response was video captured simultaneously with a stopwatch, to allow subsequent frame-by-frame extraction of the response amplitude. The time resolution on the stopwatch was 0.1 s, but due to the time required for time domain data generation on the network analyzer, the images only updated every few stopwatch intervals.

3. Results and discussion

3.1. FTIR characterization of the films

FTIR determination of water vapor adsorption–desorption behavior of the films was performed by measuring IR absorbance, in the OH-stretching vibrations of H_2O, focusing in on the
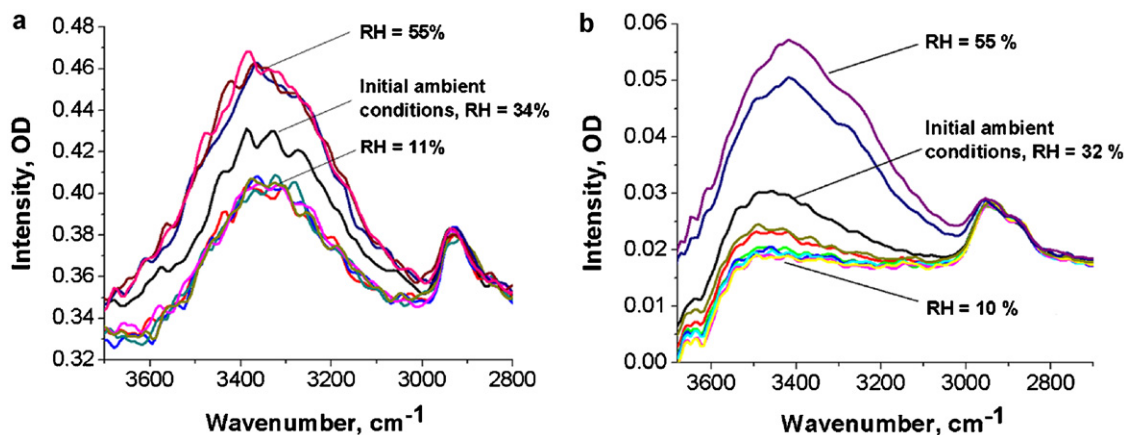
Fig. 2. Transmission FTIR-study of the thin films upon varying relative humidity: PVA-film (a) and PVP-film (b). Spectra were recorded against a bare glass as a background. Several cycles of humidity exposure were applied.

$3200–3500\,cm^{-1}$ range. The intensity of the IR absorption depends on the amount of water molecules present in the films. Thus, varying ambient humidity conditions and simultaneously measuring the intensity of OH-stretching vibrations reveals the relative water loading in the films versus RH value in the measuring chamber.

The velocity of surface acoustic wave propagation depends primarily on three parameters: mass-loading (i.e. the mass of an extraneous compound absorbed in the near surface layer), the viscoelastic loading (i.e. change in the film viscosity and/or elastic properties) and electrical loading (change in film conductivity) [9]. In the present case, at least one parameter, mass-loading, was expected to change upon water vapor absorption, although viscoelastic loading may contribute as well. The contribution of electrical loading seems to be unlikely due to the non-conductive nature of the polymer. Given this relation, IR measurements of intensity changes in the OH-stretching vibration spectral region is a suitable way of testing polymeric films in order to determine their applicability in SAW sensor technology.

In our FTIR tests we pursued two primary goals: to investigate the possible reversible character of water adsorption–desorption behavior of PVA and PVP films upon varying ambient RH conditions and, more importantly, to estimate the dynamics of this process (i.e. time required for reaching full saturation of the polymer with water vapor, and reversible process of complete desorption). The results of transmission FTIR study of PVA and PVP films upon varying humidity are shown in Fig. 2.

In both cases a broad peak is observed around $3400\,cm^{-1}$, corresponding to the OH stretching vibration of the absorbed water molecules. In addition, CH-stretching vibrations are observed between 2850 and $3000\,cm^{-1}$. Upon changing the RH in the chamber the intensities of the OH peak undergo changes. Starting from the initial humidity level, corresponding to ambient humidity in the laboratory (RH around 36%), and rapidly increasing the humidity up to about 55%, a notable increase in the peak intensity is observed (Fig. 2). Switching the gas flow to dry N_2 leads to a decrease in the IR intensity, reaching a value below the initial starting point. It should be noted that in the case of PVA, which contains OH functional groups connected to every other carbon atom in the chain, the signal at $3400\,cm^{-1}$ is a result of the overlapping of $OH_{polymer}$ and OH_{water} vibrations. This explains the remaining intensity of the peak in the case of PVA film even after drying the chamber to as low as 2% RH, when only the $OH_{polymer}$ vibration is observed. In the case of the PVP film, which contains ketone functionality rather than OH groups, one can observe almost complete disappearance of the OH peak at $3400\,cm^{-1}$ in dry conditions. Successively increasing and decreasing RH in the chamber revealed the reversible behavior of the water adsorption/desorption process for both PVA and PVP

films. The response time can be seen by plotting maxima of the OH absorption peak versus time (Fig. 3).

In order to estimate the response and recovery time of the films, the IR spectra were recorded every 4 s with simultaneous cycling of the humidity level from low (~5%) to high (~55%). It was determined that the intensity of the OH vibration reaches its maximum within 4 s for both films, i.e. the IR response of the films is on the order of several seconds. The recovery to low humidity levels takes longer time for the PVA film, requiring at least 3 spectra to be collected before reaching the initial intensity. In the case of the PVP film the time required for complete recovery of the signal was less than 4 s (one collection period).

3.2. AFM characterization of film thickness

Film thickness is an important parameter that should be controlled for optimizing SAW sensors. For instance, it was shown [9], that humidity induced jumps in attenuation and turnaround in frequency shifts of poly(XIO) coated SAW devices at 22.5 °C, due to the change in film composition causing a transition from the elastic to the rubbery state, are dependent on film thickness. Overall particle displacements in polymeric films result from the superposition of two shear waves and one compressional wave generated by the SAW at the film/substrate interface. As these displacements propagate further into the film, in-plane and cross-film particle displacements within the film can lag one another in phase, with phase shifts for each wave being related to the viscoelastic prop-
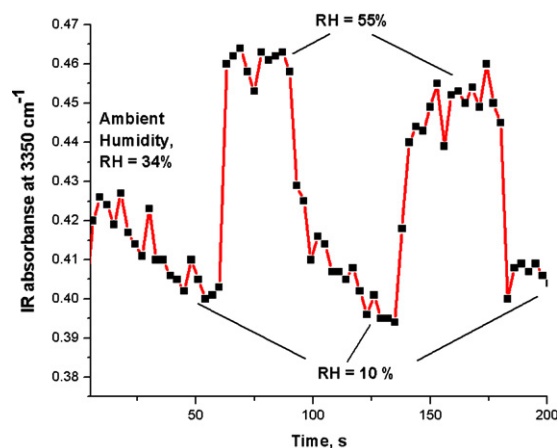

Fig. 3. Plot of the maxima of the IR adsorption at $3350\,cm^{-1}$ for PVA film as a function of time in the course of changing humidity. Spectra were collected every 16 s.
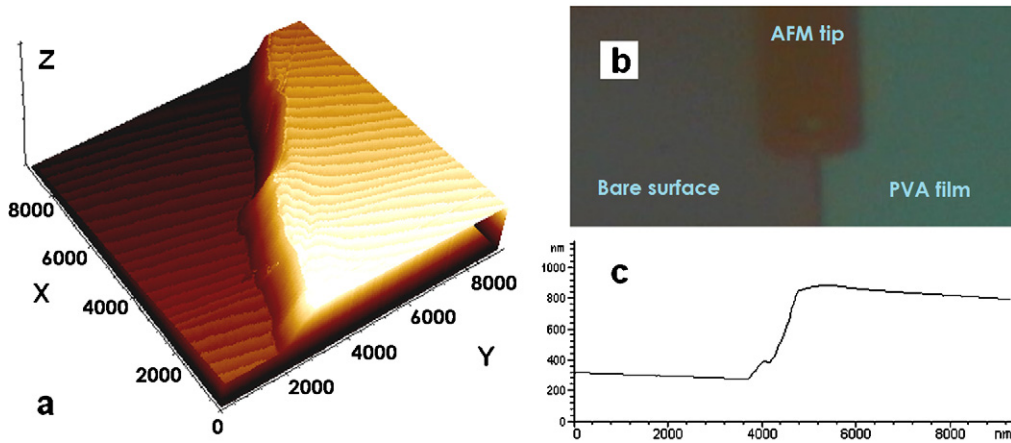
Fig. 4. (a) Typical AFM image used for film thickness measurements (on an example of PVA deposited at 7000 rpm); (b) visual microscopy image of the AFM tip positioned at the edge of the film; (c) cross-section showing profiles of the film edges.
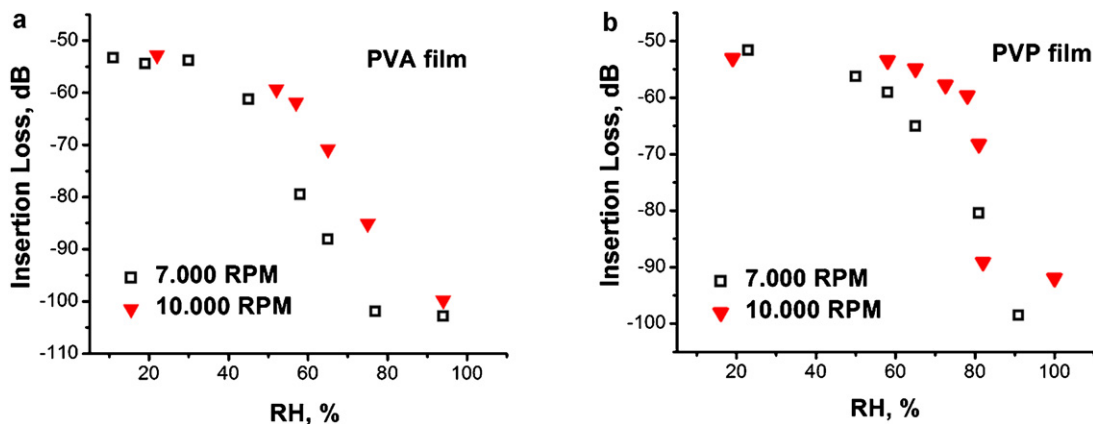

Fig. 5. SAW response (insertion loss) of PVA-based sensors (a) and PVP-based sensors (b) as a function of ambient humidity level.

erties of the film [15]. The significance of each of the component waves in terms of the effect it has on sensor performance depends on the film thickness relative to the acoustic wavelength at the frequency of operation.

Film thickness estimation was carried out by scratching the films on the actual SAW device in several different spots across the surface with a razor blade to obtain trenches, followed by AFM imaging of one of the edges of the obtained trenches (Fig. 4). Each trench was imaged by AFM in several different spots along the surface. The AFM tip was positioned over the scratch edges with help of a visual microscope, embedded in the AFM equipment (Fig. 4b; see also Figs. S3 and S4). The results of the thicknesses determined for the films, deposited at various spinning rates, are summarized in Table 1. As expected, the higher spinning speed resulted in the formation of thinner films due to more effective spreading of the polymeric material across the surface of the substrate.

3.3. Characterization of the SAW sensor response

The first two peaks in the simulated response for the device (Fig. 1b) correspond to the direct and triple transit responses of the two transducers. The second pair of peaks between 4 and 5 μs

Table 1
Results of thickness measurements using AFM imaging.

Sample	7000 rpm	10,000 rpm
PVA	660 ± 20 nm	510 ± 35 nm
PVP	200 ± 15 nm	150 ± 15 nm

corresponds to the reflections of the SAW from the reflector. The SAW propagates under the deposited polymeric film, bounces off the reflector, and propagates back under the film to the two transducers in order to generate this response. The device was designed so that this reflected response would be well separated in the time domain from the transducer responses, providing a sensitive tool with which to observe and measure changes introduced in the delay path during sensor operation. The experimental SAW response for the uncoated device (Fig. 1c) shows reasonable agreement with the simulation. The increased signal seen in the device response near $t = 0$ is RF feedthrough commonly seen in SAW devices when probed on a die level.

The SAW devices coated with PVA and PVP films, deposited at 7000 and at 10,000 rpm, were tested at room temperature with humidity ranging between 5 and 95% RH (Fig. 5). Both films showed strong SAW signal variations with changing relative humidity. The response of the thicker PVA film device drops by over 50 dB over the range of humidity change (Fig. 5a). Comparison of the magnitude of the SAW response to relative humidity for the two PVA films (Fig. 5a; see also Fig. S1 of the Supporting information) clearly shows that the difference in film thickness affects the response signals, as the SAW response drops off at a lower RH % for the thicker film (7000 rpm). This may be either due to mass loading, changes in film elastic properties, or both. Since these films are relatively thick (500–600 nm), it should be possible to produce sensors with high sensitivity for low RH levels using much thinner films. Additional work will be required to optimize these films for such specific uses. Nevertheless, this indicates that an appropriate film thickness can be selected to provide sens-
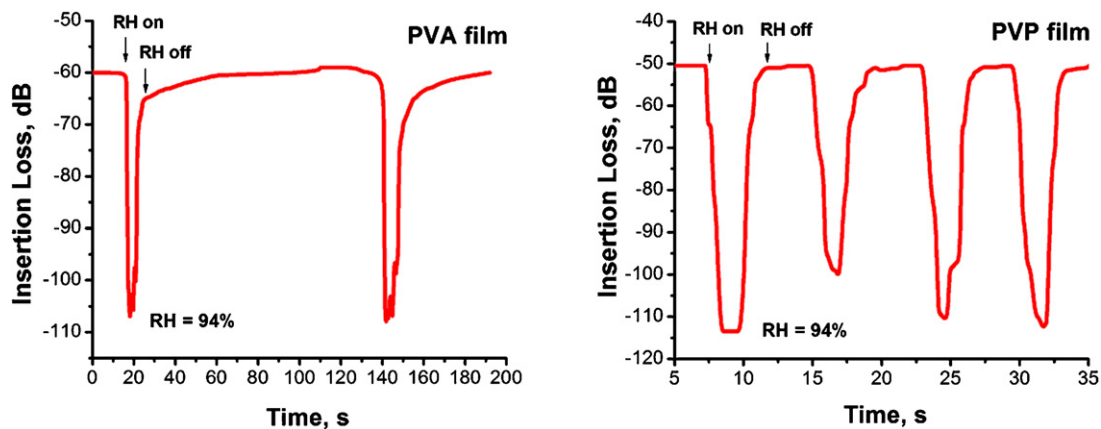
Fig. 6. SAW response dynamics of PVA-based sensor (a) and PVP-based sensor (b), measured for a step from 5 to 94% RH.

ing capability over the humidity ranges of interest for particular applications.

Humidity tests were also conducted for PVP coated SAW devices, with results shown in Fig. 5b. Similarly to PVA-based sensors, the PVP films showed a difference in SAW response to variation of RH% for the two films studied, with a larger response for the thicker films. It is shown that the thicker film is capable of discriminating RH from 20% to almost 95% with good resolution, while in the case of thinner PVP film the sensor has lower humidity resolution (Fig. 5b; see also Fig. S2 of the Supporting information). Note that at about 80% RH, the thinner PVP film exhibits an increase in insertion loss of about 15 dB, while the thicker film showed more than 30 dB increase. Thus film thickness is a key factor to set sensor resolution. It is interesting to note that for the thinner (10,000 rpm) PVP film, there appears to be an abrupt jump in insertion loss that occurs at a humidity level of about 83%. This may be due to a transition of the polymer film from the glassy (elastic) to the rubbery (elastomeric) state. Such a transition should occur at the glass transition temperature, which for bulk PVP (from BASF) is about 150 °C. However, absorption of water by the polymer film effectively changes the film composition, and should change the glass transition temperature accordingly. Tashtoush and colleagues observed this glassy to rubbery transition in PolyXIO coated SAW humidity sensors at 22.5 °C [9]. While such an effect has not been reported previously for PVP coated SAW sensors, it may be responsible for the observed sudden large drop in response. The thicker PVP film showed better SAW response resolution for humidity measurement over a broad RH range (Fig. 5b) compared to the thinner films deposited at 10,000 rpm.

Although direct comparison of the PVA and PVP films is not appropriate in this work because of different deposition conditions, e.g., different solvents and polymer concentration, one can observe some general tends for the two films as a function of their thickness. In the case of PVA, the thicker film was about 660 nm, while the thinner film was about 510 nm. The thicker film could be used to produce highly sensitive humidity sensors capable of operating over a RH range restricted to ambient (about 36%) to 75% RH. Even thicker films might be required to achieve sufficient humidity resolution at very low RH, albeit with somewhat slower response times. The thinner PVA film shows better capability of measuring humidity over a higher range of RH values ranging from ambient to saturation. The PVP films tested were considerably thinner, with the thick PVP film measuring 200 nm and the thinner PVP film measuring 150 nm. The thicker PVP film showed large, measurable response changes for a fairly wide range of RH levels, from ambient to saturation. The thinner PVP film appears more sensitive to low RH levels, being capable of responding to levels below ambient humidity (see 19% RH curve in Fig. S2,b, Supporting information),

but is less responsive to changes at higher RH. The rapid drop in SAW response above 83% RH is likely due to the transition of the PVP film from a glassy to rubbery state as more water is absorbed into the polymer film, effectively changing the film composition. It can be seen that the sensitivity of the SAW sensor to RH changes can be modified by controlling the characteristics and thickness of the sensitive polymer film. Thus, for each particular polymer considered, and for each desired RH operating range, film thickness should be evaluated and adjusted in order to establish the optimal film thickness value to meet the sensitivity requirements in that RH range. It should be noted that sensitivity of both films to relative humidity is quite large compared to previously reported SAW vapor sensors based on polymer films, which generally exhibit smaller insertion loss changes of up to 5 or 10 dB for RH exposures of up to 90% [17,18].

The response time against change in humidity from 5 to 95% RH was measured for both PVA and PVP-based sensors. The network analyzer plot of the device response was captured simultaneously with an electronic stopwatch, to allow subsequent frame-by-frame extraction of the response amplitude as a function of time. The stopwatch time resolution was 0.1 s, but due to limited time domain data generation refresh rate of the network analyzer, the images are only updated every few stopwatch intervals. The speed of the network analyzer computations thus limited our ability to observe response times to the order of at least 0.4 s. The response of the PVA film deposited at 10,000 rpm to humidity was rapid (within 2 s) and the recovery took longer period of time, being about 5 s for 80% recovery of the signal with a long "tail" of additional 35 s until full recovery (Fig. 6a). The PVP deposited at 10,000 rpm responded even more rapidly showing response and recovery times around 1.5 and 2.5 s, respectively (Fig. 6b). Overall, PVP-based sensor appeared to have better sensor characteristics over its PVA analog. Compared to thinner PVP analogues, thicker PVA films exhibit substantially longer times required for signal recovery, after dry air flow is provided (Fig. 6). This is probably due to the larger amount of water absorbed by thicker films, and, as a result, the longer period of time needed for water to leave the film. This is evidencing that thickness of the film is a parameter which influences response time. Another reason is more pronounced hydrophobic nature of PVP polymer compared to PVA that causes weaker water molecule bonding to the polymer surface and, as a result, more "flexible" response behavior.

In order to better characterize response dynamics of the PVP-based humidity sensor, comparative study was conducted aiming to relate performances of the studied sensor and commercial device (Omega RH-32B) used for controlling humidity levels in our work. Several features of the responses shown in Fig. 7 are noteworthy. First, it is seen that the studied SAW sensor responds faster (within 1.5 s) than the commercial sensor, which is rated to have
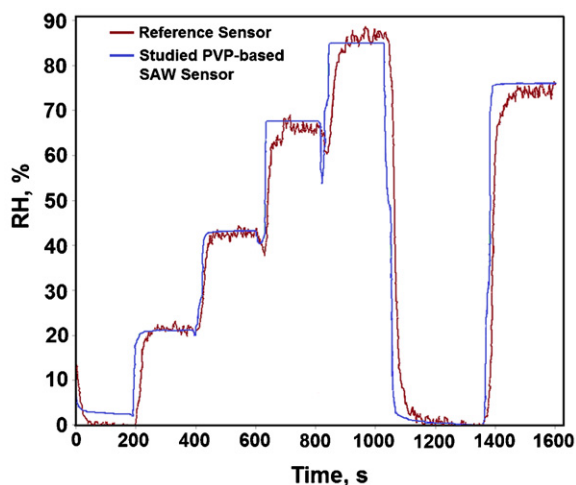
Fig. 7. Comparative study of the sensor performance for the SAW humidity sensor, designed in this work, and commercially available device.

a 5 s response time, and was one of the fastest sensors available at the time of purchase. Second, we note that the SAW response has a very low noise level compared to the commercial sensor. Data was acquired from both the SAW device and the commercial sensor once per second. Noise on the order of 3–5% RH is observed for the commercial sensor, while the SAW sensor (neglecting drift) shows very little noise. This may in part be due to the fact that a network analyzer, which is capable of very low noise measurements, is being used to take the SAW data. Realistic performance in an electronic reader might exhibit more noise, however, it is beneficial that the sensor itself does not exhibit significant noise mechanisms. Third, it is interesting to note that this sensor responds in a very nearly linear fashion, with the raw SAW sensor data scaling almost directly to the actual relative humidity level (as measured by the Omega sensor). The obtained data shows apparent improvement of the sensor characteristics for the studied SAW sensor over commercial counterpart.

4. Conclusions

Transmission FTIR study is a suitable method of quick screening polymer films for their applicability for fabricating SAW humidity sensors. In particular, the response and recovery dynamics of potential SAW device can be roughly estimated as a result of such IR measurements without the need to incorporate the films into actual SAW system.

Thin films of PVA and PVP polymers were deposited onto SAW substrates using spin coating. The thickness of the obtained films was controlled in the region of several hundred nm as estimated using AFM imaging. The SAW response of the obtained sensors was measured upon varying humidity from 5 to 95% RH. Both films showed high sensitivity to relative humidity and prompt response dynamics, with better parameters of the PVP-based sensor which demonstrated fastest dynamics among reported SAW humidity sensors based on polymer materials. Simplicity of the obtained SAW sensor and its prominent sensor characteristics make it an attractive candidate as a commercial wireless humidity detector.

Acknowledgements

Appendix A. Supplementary data

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