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Temperature Based Rapid Saw Humidity Sensor

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ABSTRACT

This paper investigates the effect of temperature on the sensitivity of a thin-film Polyvinyl Alcohol (PVA) based SAW humidity sensor. A PVA coated 433.92 MHz SAW resonator-based humidity sensor was fabricated and tested with different levels of humidity (0.5 to 95% RH) at different operating temperatures (10°C to 70°C). The sensor response was recorded through in-house developed data acquisition software and it was observed that PVA thin film coated SAW sensor shows the maximum sensitivity for trace level moisture detection at a lower temperature (≤10 °C). The sensor sensitivity has been recorded >400 Hz/% RH for trace level detection (0.5–30% RH). It has been observed that sensor sensitivity deteriorates when temperature increased to 40 °C from 10 °C. The sensor has a fast response (~1s) and recovery time (<3s) for trace level humidity detection. The proposed sensor can be used in many applications, including fabrication of electronic devices, IC fabrication, pharmaceutical, textile industries, food processing, semiconductor device fabrication, and packaging.

Keywords: PVA; Humidity; SAW sensor; Sensitivity; Operating temperature

I. INTRODUCTION

Monitoring of humidity is necessitated due to increasing concerns in the fields of industrial production, respiration monitoring, food storage, agriculture, meteorology, environment, chemical synthesis, pharmaceutical, etc. Yunpen su et al. studied and proposed SAW delay line-based humidity sensor by using three-dimensional graphene/ PVA/SiO2 material. Xinyu Zheng et al. used the combination of a thin film of nano silica and PVA as composite material on Quartz Crystal Microbalance (QCM) for the detection of humidity and also studied the effect of coating thickness of PVA alone, different ratios of the mixture of PVA with nano silica materials[1-2]. Xian hao le et al. reported AlN/Si-doped layer and graphene oxide as interface material for detection of a wide range of humidity from 5 %-95 % RH and also reported that after coating the composite material on QCM, the thermal coefficient (TCF) of the device improved to -22.1ppm/°C[3]. U. Mittal, *et al*. reported γ-Al_2O_3 thin film based SAW humidity sensor prepared by sole Gel method for measurement of moisture in the range of 3 %-85 % RH[4].

The increased humidity levels many a time create unavoidable problems for the system, which hampers the performance of the system. In literature, it is evident that researchers have developed many types of humidity sensors, such as SAW, capacitive, resistive, field effect transistor, quartz crystal microbalance[1, 5-13] etc. Moreover, some other types of sensing materials i.e. polymers, metal oxide, carbon nanotubes, composite material[2,4,13-16] etc. have also been actively used in the fabrication of humidity sensors. Piezoelectric materials-based SAW devices are also used for the fabrication of chemical sensors due to high sensitivity to physical parameters i.e., perturbation /or its associated electrical field [17]. The thin film is used to convert the value of the desired parameter into a mechanical or electrical perturbation that can interrupt the SAW velocity causes the potential of the sensor[4, 18-19]. Mostly three mechanisms are actively involved in the SAW sensor response: (1) Mass-loading, (2) Acoustoelectric, and (3) Viscoelasticity[20] as shown in Eqn. (1).

$$\frac{\Delta F}{f_0} = -C_m f_0 \Delta(\rho_s) + C_e f_0 h \Delta\left(\left(\frac{4\mu}{v_0^2}\right) \times \left(\frac{\mu+\lambda}{\mu+2\lambda}\right)\right) - \frac{K^2}{2} \times \Delta\left(\frac{1}{1+\left(\frac{v_0 c_s}{\sigma_s}\right)^2}\right) \quad (1)$$

where mass sensitivity and elasticity coefficients of the substrate are represented as c_m and c_e respectively, (*m/A*) is the mass change per unit area, f_0 and h is the fundamental frequencies of device and film thickness respectively G' and k^2 is the storage modulus and electromechanical coupling coefficient and σ_0, c_0 and v_0 is the conductivity, capacitance, and velocity of the SAW device. The basic structure of the SAW device having two metallic inter digital transducer (IDT) on the surface. The IDTs are made of conducting material for which gold is preferred due to high conductivity, adhesiveness, and anti-corrosion property. The device used in the proposed work uses aluminum-based IDTs, due to low cost, comparable conductivity and ease of fabrication, schematic of SAW device is shown in Fig. 1.

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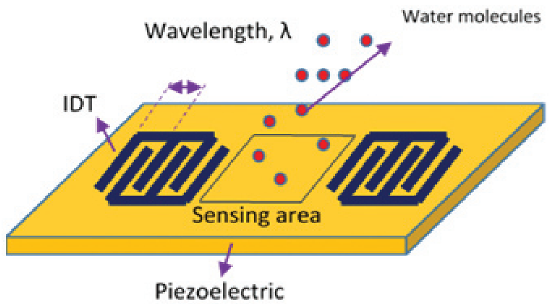

Figure 1. Schematic of SAW device

SAW-based sensors are most desirable as compare to the other conventional sensors due to high sensitivity, low cost, small size, wireless operation[18,20,21-24] etc.. In the literature, the researchers have mostly relied on SAW resonators for the fabrication of sensors due to their high Q factor and narrower bandwidth. Generally, PVA thin film coated resonators were used for the fabrication of humidity sensor due to their lesser noise, higher sensitivity, and low drift as compared to the other coating material[4, 23, 25-30]. In literature, the sensing mechanism of SAW sensors is explained based on surface perturbations due to film, mass loading, elasticity, temperature, pressure[18,31–36], etc. PVA material is an excellent material for SAW humidity sensor due to its excellent hygroscopic properties, functionality with -OH group and higher molecular weight makes it an excellent choice for the SAW-based humidity sensor. The sensitivity of the SAW device changes drastically with operating temperature.

A novel work has been carried out for the study of the effect of operating temperature variation on PVA based humidity sensor. As per the best of our knowledge and available literature, the researcher has not studied the performance of PVA based SAW humidity sensor considering this aspect. This study is focused to establish the sensor performance for detection/measurement of humidity in extreme thermal conditions. The study proposes a stable dual-mode SAW-based sensor for accurate measurement of humidity at trace to a high level under various operating temperatures. Since the frequency of SAW drastically varies due to change in the operational temperature, therefore this study also provides excellent information for tuning the sensor as per the environmental condition. It described the effect of temperature and thickness optimization of PVA thin film for fabrication of SAW based humidity sensor.

2. EXPERIMENTAL WORK

2.1 Preparation and Characterization of Thin Film of PVA

A solution of PVA was prepared by dissolving PVA in deionized water with different ratios (1 to 10mg/ml) and subsequently stirred the solution for 3 hours at 70°C, thereafter 0.5 microliter sample of each solution was coated on the SAW device and cured 4 hours at 80°C in the oven. Subsequently, all coated devices were tested with 50% humidity and observed that those devices coated with 5 mg/ml PVA solutions showed very good sensitivity response. It was also observed that the devices which were coated with more than 5mg/ml solution could not operate in oscillator configuration due to higher mass loading of PVA on the surface of the SAW device. The response of coated device with less than 5 mg/ml PVA solution was not encouraging (Fig. 2a). It was also observed that PVA coating also acts as a passivation layer for inter digital transducer. To study the morphology of thin film, same PVA solution (5mg/ml) coated on the plasma cleaned quartz wafer and kept to dry at 80°C for 4 hrs. The roughness of the film surface is studied by atomic force microscopy (AFM) (model 5600LS of Agilent Technology), which shows a very good uniformity (Fig. 4b).

2.2 Fabrication of SAW Oscillators

To carry out the testing of PVA coated device, a one port SAW resonator of 433.92 MHz was fabricated in dual mode oscillator configuration[39-40]. Sensor assembly contains two SAW devices, one uncoated act as a reference and another PVA coated used as a sensor. In-circuit a mixer was used for obtaining the difference signal frequency of sensor and reference devices. To avoid any other physical parameter effect (temperature, flow, pressure etc.) on reference device, used in a TO-39 package. The sensor device is placed on the thermoelectric cooler (TEC) directly which can operate at desired temperature of 10 °C to 70 °C quickly by changing the operating voltage. The covered device was operating at room temperature, which was not affected by flow, temperature, and pressure change. Hence the sensor is significantly stable as compared to the open device. A Printed Circuit Board (PCB) along with the sensor cell is shown in Fig. 3.

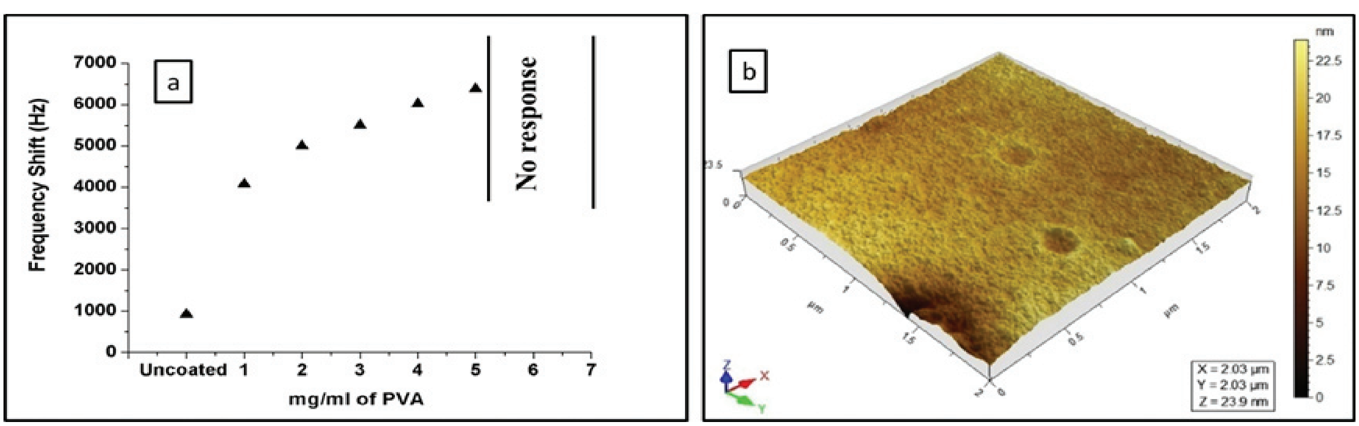

Figure 2. (a) Frequency change of the SAW with a different PVA solution and (b) AFM of PVA film.

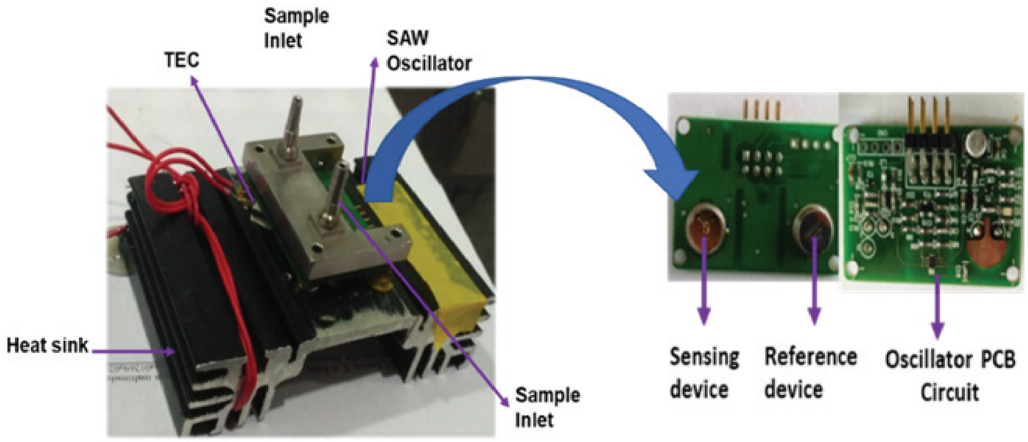

Figure 3. Sensor cell with TEC, heat sink and Oscillator PCB with SAW devices.

2.3 Assembly and Characterization of SAW Humidity Sensor

The SAW device surface was clean using oxygen plasma for removing the unwanted impurity before the coating of PVA. The PVA solution was coated on the full surface of the sensing device along with the interdigital electrode by drop dry method and cured at 80°C for 4 hrs in the oven. The response of the SAW- PVA based humidity sensor depends on the thickness of PVA film, uniformity of the film, operating frequency, the noise of the oscillator (in case of trace level detection), etc. The thickness of the PVA film has been optimized for better sensitivity using the iterative method.

2.4 Experimental Setup

The different level of humidity was generated by humidity generator (Owlstone OHG-4). A high purity [99.999 %] dry nitrogen passed through a water tank where the bubbler generates the desired humidity in the range of 0.5% to 95% RH. The humidity level is controlled by varying the flow of dry nitrogen along with wet flow through the water tank[39]. The same humidity generator system was upgraded with a switching setup which provides the alternating switching between the sample of humidity and dry nitrogen gas to the sensor cell. In 'ON' condition it passes the humidity (wet nitrogen) through the sensor cell and at the same time dry nitrogen vent out through the vent and in 'OFF' condition it passes only dry nitrogen through the sensor cell and at the same time humidity vent out through the vent. The sensor is attached with this alternating switching setup for testing with different humidity levels. A standard humidity sensor (DTH10 RH meter) was also used for confirmation of generated humidity at the output port of the sensor. The schematic humidity generator and actual test setup is shown in Fig. 4.

The change in frequency of SAW was measured through a data acquisition system on a computer. Experiments were conducted to examine the sensor parameters, i.e. (1) frequency responses for 0.5 %–95 % RH at different temperatures; (2) recovery and transient response times.

3. RESULT AND DISCUSSION

It is evident that the sensitivity response of the sensor rises with higher humidity levels. It was observed and achieved the significant response of PVA coated SAW humidity sensor for trace level detection (0.5-30 % RH) at ≤ 10 °C (Fig.5b). During the experiment it was found that maintaining the temperature at ≤ 10 °C and increasing the RH > 40 % makes sensor non-responsive, subsequently, the temperature of the sensor was increased in the steps of 10 °C and it was found that the sensor

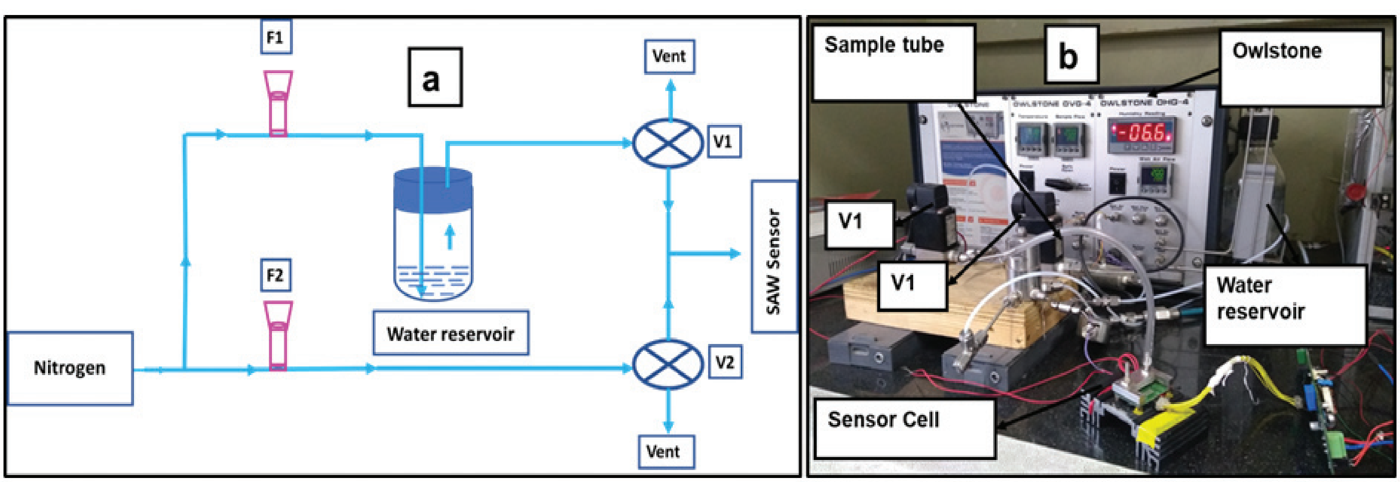

Figure 4. (a) Schematic of the humidity generator setup and (b) Actual test setup of humidity generator.

operating temperature of 40 °C is the most suitable temperature to measure the entire humidity range from 1 % to 95 % RH (Fig.6). The sensor can be made to have two modes viz. one to operate at ≤ 10 °C (for trace level detection of humidity) and the other operated at 40 °C. Since the operating temperature of 40°C facilitates sensing the entire humidity range from 1 % to 95%, therefore, the performance comparison between PVA coated and the uncoated devices was done at 40°C. It was observed that the overall frequency response of the coated device for humidity range from 40 % to 95 % RH is nearly exponential and from 1 % to 40 % shows linear behavior at 40 °C. However, the uncoated device shows the linear behavior for 1 % -95 % at 40 °C as shown in Fig. 5a. and can be described by the following relation[4]:

$$In\left(f - f_0\right) = a\left(RH - RH_0\right) + b$$

Where f_0 is the resonant frequency, RH_0 reference of relative humidity, *a* and *b* are the coefficient constant.

Subsequently, sensor tested with different humidity at different operating temperature (10 °C to 70 °C) and it was observed that the water molecules adsorbed more effectively on the surface of the SAW device at a lower temperature (≤10°C) and high humidity which makes sensor non-responsive due to excess of mass loading of water molecules on the surface of SAW device Fig. 6(a). It was also observed that when the sensor was exposed to a humidity level of <0.5% RH at 10°C the water molecules in vapor form adsorbed on the surface of the device in nano-order layer, causing frequency change due to mass loading effects providing measurable frequency response. When dry nitrogen gas passes over the surface of the SAW device it desorbs the water molecules from the surface of the SAW device. The sensor shows the fast response time (~1s) and fast recovery time (<3s) for trace (0.5 %RH) level sensing of humidity at 10 °C as shown in Fig 6b. The sensor does not show a significant signal towards the trace level (<1 % RH) humidity at 40 °C. Hence, for the trace level detection of humidity, the sensor should operate at 10 °C instead of 40 °C.

The SAW frequency changes due to the changes in capacitance and conductance of the sensitive film on the surface of the SAW device, but it is significantly less as

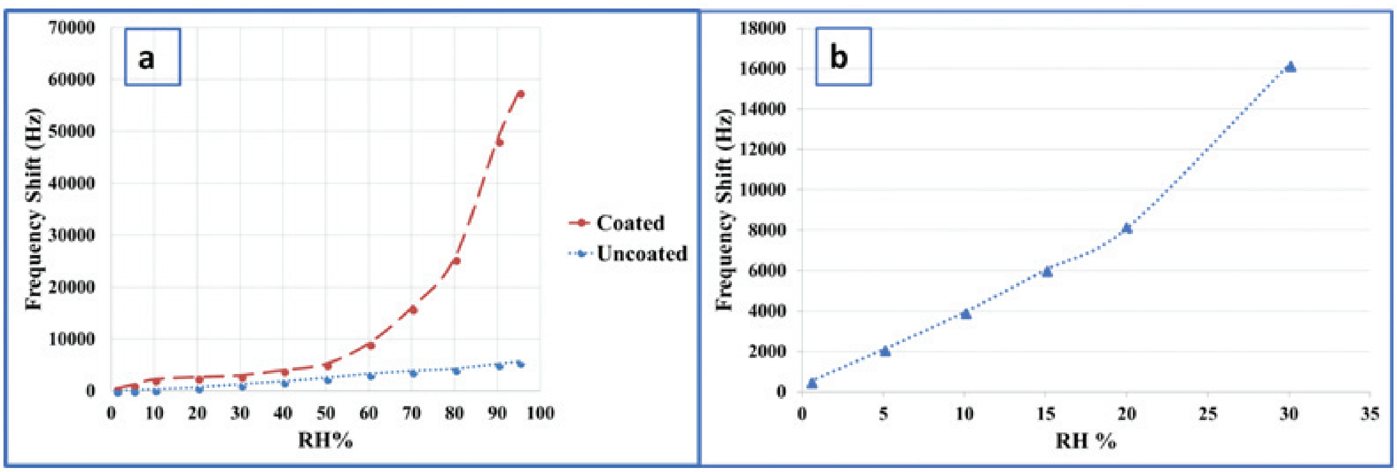

Figure 5. (a) Sensor response with coated and uncoated device at 40 °C with humidity range 1 % to 95 % RH and (b) response at 10 °C with trace level humidity range 0.5 to 30 % RH.

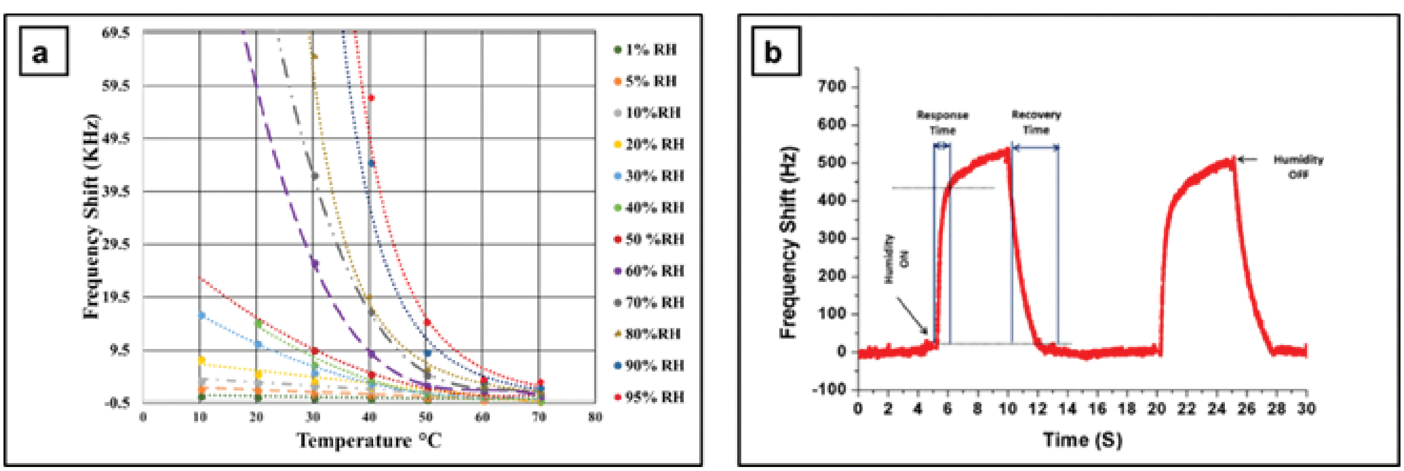

Figure 6. (a) PVA coated SAW sensor response with humidity range of 1 %–95 %RH at different operating temperature and (b) Sensor response with 0.5 % RH at 10 °C.

compared to the mass loading[4]. Initially, water molecules are attached to the active sites of PVA -OH groups with hydrogen bonding due to its hygroscopic nature of PVA film and gives the mass loading effect[40]. Due to the easy path of proton hoping with adsorbed water molecules, the capacitance may increase as well as conductance may also increase due to the increase of proton (H+) concentration on the surface of the PVA film[26, 40]. It was observed that the sensor shows very less hysteresis error (<0.3 %) from 0.5 % to 30 %RH due to the good stability of thin-film[41-42]. The drift behavior of the SAW sensor was also studied and observed that after three months sensor shows negligible drift. The morphology of film plays a vital role in the adsorption of water molecules on the surface of the film. The sensitivity of the SAW sensor also depends on the operating frequency, nature of thin-film, and mass-loading mechanisms[4, 43]. The linear response of the sensor for trace level detection shows its high potential for detection of trace level (<0.5 % RH) of humidity at10 °C temperature.

4. CONCLUSION

In this paper, a thin film PVA based SAW humidity sensor was fabricated. The sensitivity of the sensor was studied at different temperature range (10°C to 70°C) and relative humidity (0.5 %-95 %RH), the sensitivity of the sensor was enhanced at low temperatures for the detection of trace humidity by condensation of water molecules on the surface. The PVA film was used as interface material for humidity sensing as well as a passivation layer for SAW device IDTs. The sensor response for 0.5 % to 95 % RH at 10 °C to 70 °C temperature has been studied and it was observed that the sensor response at low temperatures for the detection of trace level humidity (0.5 % RH) was > 400Hz /%RH. It is also found that sensor is highly reproducible in behavior and has a fast response and recovery time. It can be used as a dual mode-based humidity sensor for accurate measurement by switching the mode. One mode can be used for trace detection of moisture (at low temperature) and another mode can be used for > 30% RH (at 40 ºC).

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