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Measurement of Moisture Absorption Ratio of FRP Using Micro Polymer Sensor

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Abstract. Moisture absorption of FRP is important because the mechanical properties, such as compressive and fatigue strength, significantly degrade due to moisture absorption. It is difficult to apply conventional measuring methods to FRP because of its inhomogeneity and low moisture absorption ratio. In this study, a new method to measure the moisture absorption ratio of FRP by the change of the dielectric constant, which corresponds to the capacitance, of a micro polyimide sensor embedded in FRP is proposed. At first, the relationship between the moisture absorption ratio and the dielectric constant change of the sensor itself is investigated. Then, GFRP specimens in which the sensor is embedded in the midplane are immersed in water, and the moisture absorption ratio of the specimen and the dielectric constant change of the sensor are monitored. As a result, it is shown that the moisture absorption ratio of GFRP can be easily measured by the proposed method.

Introduction

Recently applications of fiber reinforced plastics (FRP) have increased in many structures, e.g., marine structures and wind turbines. In natural environment, FRP structures absorb moisture because of existence of humidity in atmosphere and rain. Since the absorbed moisture adversely affects the property of matrix and matrix/fiber interface, the mechanical properties, such as compressive and fatigue strength, and glass transition temperature significantly degrade [1]. Measurement of moisture absorption ratio of FRP is important, but difficult by conventional methods because of its microscopic inhomogeneity and low moisture absorption ratio at most 1.5%wt for epoxy resin.

In this study, a new measurement method of the moisture absorption ratio of FRP by a micro sensor made out of a thin polyimide film is proposed. Electrode pattern is spattered on the both side of the film and the dielectric property is measured to monitor the moisture absorption ratio of the polyimide film. The sensor is embedded in FRP laminated structures, and the moisture absorption ratio of FRP is indirectly measured. Because the dielectric constant of water is much higher than that of polyimide, the dielectric constant of the sensor significantly changes by a little moisture absorption. Since polyimide is thin, flexible and heat resistant thermoplastic polymer, the sensor can be embedded in FRP laminates. In addition, polyimide has been used for the material of humidity sensor [2-4], and thus there are many experimental and theoretical investigations about moisture absorption of polyimide [5-13]. At first, the relationship between the moisture absorption ratio and the dielectric constant change of the sensor itself is investigated. Then, GFRP specimens in which the sensor is embedded in the midplane are immersed in water, and the moisture absorption ratio of the specimen and the dielectric constant change of the sensor are monitored. As a result, it is shown that the moisture absorption ratio of GFRP can be easily measured by the proposed method.

Sensor for moisture absorption

Principle of sensor. Polyimide film for moisture absorption sensor is Kapton® which is made by DuPont©. A Film is cut into 25×20(mm), and gold electrode patterns are spattered on both sides of the film using Sanyu Electron Co.,Ltd. QUICK COATER SC-701 as shown in fig.1.

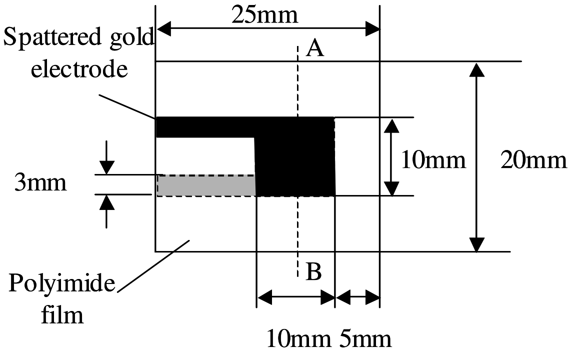


Fig. 1. Configuration of moisture absorption sensor

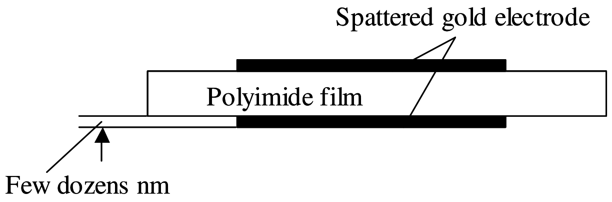


Fig. 2. A-B cross section in fig.1

A-B cross section in fig.1 is shown in fig.2. The 10×10(mm) section in the center of the sensor forms a capacitor. Capacitance of this sensor is calculated by the following equation.

$$C = \varepsilon_0 \varepsilon \frac{S}{d} \tag{1}$$

where ε_0 is the dielectric constant in vacuum, ε is the specific dielectric constant of polyimide, S is the area of the electrode, and d is the thickness of the polyimide film.

The sensor is supposed to be embedded in FRP laminated structures. By using the dielectric constant change of polyimide due to diffusion of water, the moisture absorption ratio of FRP is indirectly measured. Though it is difficult to determine the relationship between the moisture absorption ratios of polyimide and matrix surrounding the sensor theoretically, it is not unreasonable to suppose that their moisture absorption ratios are almost the same.

The relationship between the dielectric constant of polyimide and the moisture absorption is necessary to calculate the moisture absorption of polyimide from the measured capacitance. For the calculation, many mixture lows for a system containing dispersed water in polyimide are proposed. Typical equations are shown below.

Rayleigh [10]: $$\varepsilon = \varepsilon_{PI} \frac{2\varepsilon_{PI} + \varepsilon_w + 2v_w(\varepsilon_w - \varepsilon_{PI})}{2\varepsilon_{PI} + \varepsilon_w + v_w(\varepsilon_w - \varepsilon_{PI})} \quad (v_w < 0.2) \tag{2}$$

Wagner [11]: $$\varepsilon = \varepsilon_{PI} \left[1 + 3v_w \frac{\varepsilon_w - \varepsilon_{PI}}{2\varepsilon_{PI} + \varepsilon_w} \right] \quad (v_w < 0.05) \tag{3}$$

Böttcher [12]: $$\varepsilon = \varepsilon_{PI} + 3v_w \varepsilon \frac{\varepsilon_w - \varepsilon_{PI}}{2\varepsilon + \varepsilon_w} \tag{4}$$

Looyenga [13]: $$\varepsilon = \left(v_w \left(\varepsilon_w^{1/3} - \varepsilon_{PI}^{1/3} \right) + \varepsilon_{PI}^{1/3} \right)^3 \tag{5}$$

where v_w is the volume fraction ratio of water, ε_w is the dielectric constant of water, and ε_{PI} is the dielectric constant of desiccated polyimide. The choice of an appropriate mixing low for the polyimide used in this study will be discussed later.

Moisture absorption and desorption test. In order to obtain the relation between the moisture absorption ratio of polyimide and the capacitance change of the sensor, moisture absorption and desorption tests of a 100×100(mm) film and the sensor are carried out. Thickness of the film and the sensor is 125μm.

At first moisture absorption tests are conducted. After enough desiccation of the film and the sensor more than 24 hours', they are immersed in a water bath filled with purified water at 20ºC, and moisture absorption is accelerated. Then, the weight gain of the film and the capacitance change of the sensor are periodically measured. Moisture absorption ratio M of the film is calculated by the following equation.

$$M = (w_t - w_0) / w_0 \times 100 \quad (\%) \tag{6}$$

where w_t is the weight at measured time, w_0 is the weight at desiccated state. Weight of the polyimide film is measured by Electronic Balances manufactured by Shimadzu Cooperation. Capacitance of the sensor is measured at 1kHz by using 3532-50 LCR HiTESTER with 9262 Test fixture manufactured by HIOKI E.E. Cooperation. Both measurements are carried out after wiping surface water by paper filter.

After saturation of moisture absorption, moisture desorption tests are performed. The saturated film and the saturated sensor are placed in DESK-TOP TYPE HIGH-TEMP CHAMBER ST-110B1 manufactured by ESPEC Cooperation and dried at 30ºC. The weight change of the film and the capacitance change of the sensor are periodically measured.

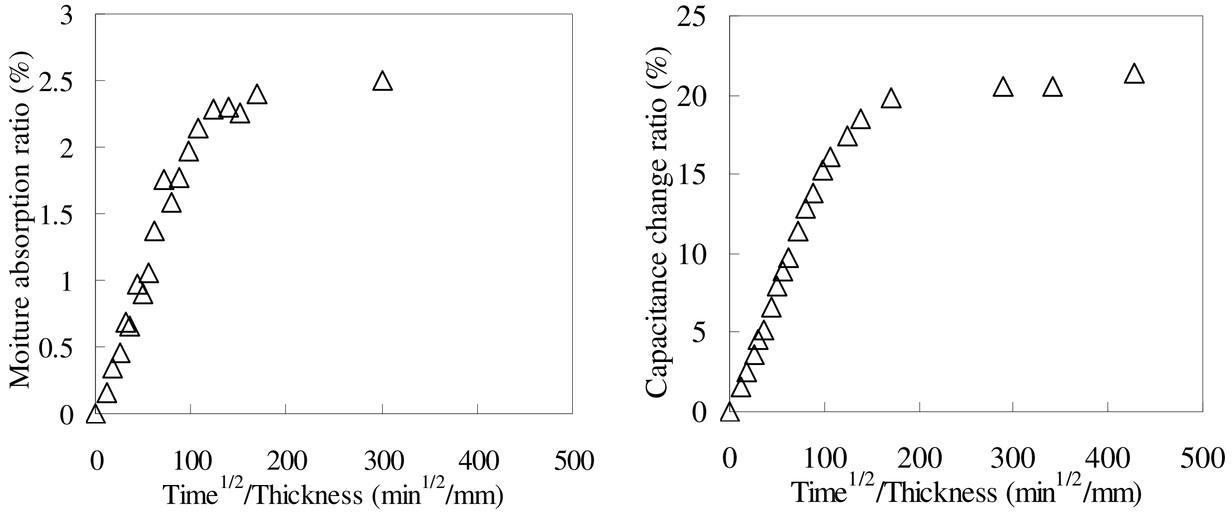
Results and Discussion. It is well known that moisture diffusion for polymer generally obeys Fickian low. In the proposed sensor, thickness of the polyimide film is thin enough to assume the infinite plate. The following relationship between moisture absorption ratio M, measurement time t and thickness of film d is derived in the initial stage of absorption and desorption for the thin and infinite plate [14].

$$M \propto \frac{\sqrt{t}}{d} \tag{7}$$

Fig.3 and Fig.4 show the moisture absorption ratio and the capacitance change ratio of the sensor for moisture absorption tests and moisture desorption tests, respectively. Fig.3 and fig.4 are plotted in terms of $\sqrt{t}/d$.

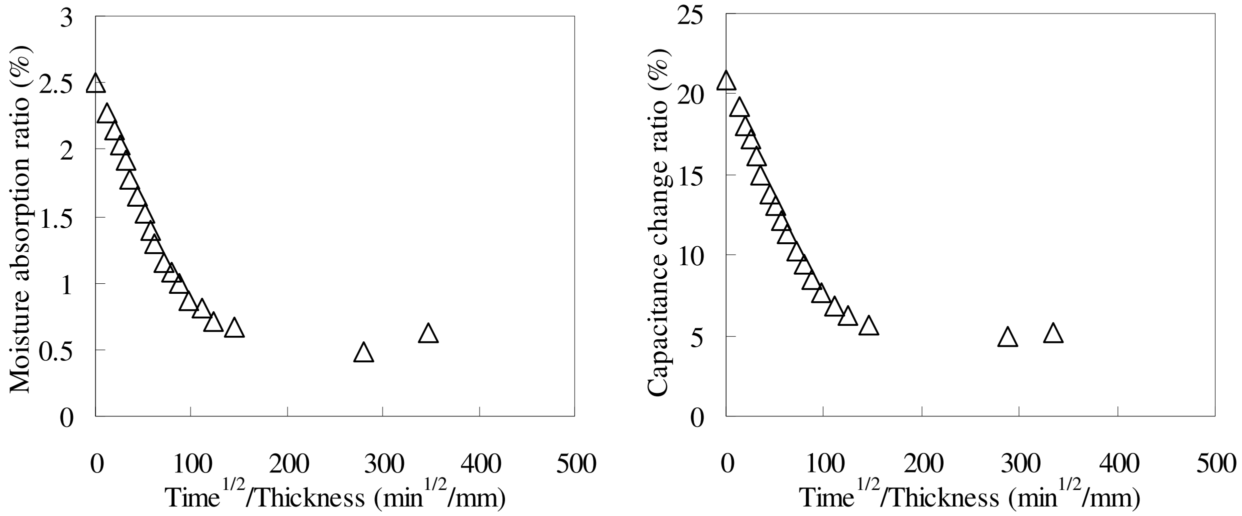
In the initial parts of moisture absorption and desorption ratio curves in fig.3 (a) and fig.4 (a), they are proportion to $\sqrt{t}/d$ and saturate at the end of experiments. The fact means that moisture diffusion in the polyimide film obeys Fickian low. As you can see in fig.3 and fig.4, the capacitance change ratio of the sensor has strong correlation with the moisture absorption ratio.

In order to determine the most appropriate mixing low, the experimental dielectric constant changes are plotted in terms of water volume fraction ratio, and compared to the mixing lows in fig.5. The ordinate is the dielectric constant change ratio, which is equal to the capacitance change ratio, and the abscissa is the volume fraction ratio of water. It is clearly shown for the material used in this study that the equation proposed by Looyenga agrees well with experimental results regardless of moisture absorption or desorption. Note that there is no arbitrary parameter in Eq.(5). These results show that we can determine the accurate moisture absorption ratio of the polyimide sensor from the measured capacitance by using Eq.(1) and (5).



(a) Moisture absorption ratio (b) Capacitance change of sensor

Fig. 3. Moisture absorption test



(a) Moisture absorption ratio (b) Capacitance change of sensor

Fig. 4. Moisture desorption test

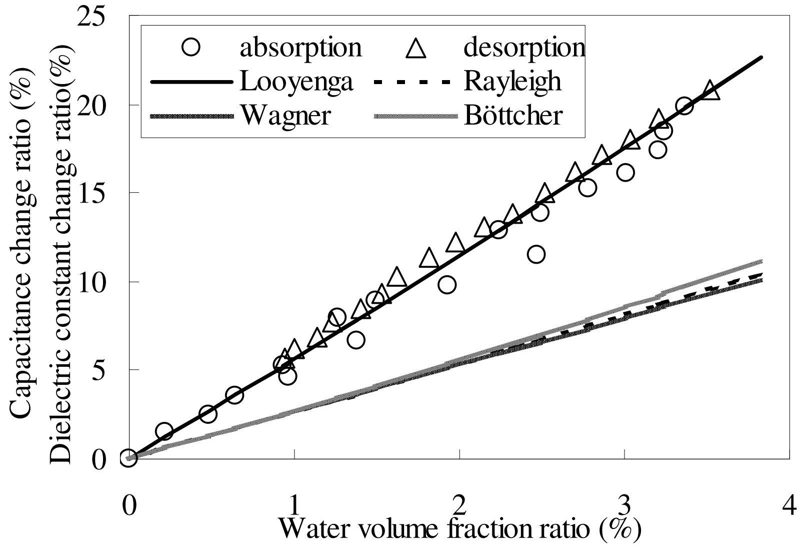


Fig. 5. Comparison between experimental capacitance change and mixing lows

Effect of thickness of polyimide film. The thinner the film is, the easier embedding the sensor in FRP is. For the reason, influence of film thickness is investigated in this section. Three films with different thickness 50, 75 and 125μm are used as the material of the sensor. Moisture absorption test and desorption test is carried out for all the sensors. In the absorption test, sensors are immersed in a water bath filled with purified water at 20ºC. In the desorption test, they are placed in desk-top type high-temp chamber at 30ºC, and the capacitance is measured at 1kHz by LCR HiTESTER. The capacitance change, which is equal to the dielectric constant change, of each sensor is compared to each other. In addition, capacitance change ratio by moisture absorption and desorption is estimated by substituting the experimental moisture absorption ratio into Looyenga's equation, and compared to them. The latter comparison corresponds to check the accuracy of the estimation from the capacitance change to moisture absorption ratio by the equation.

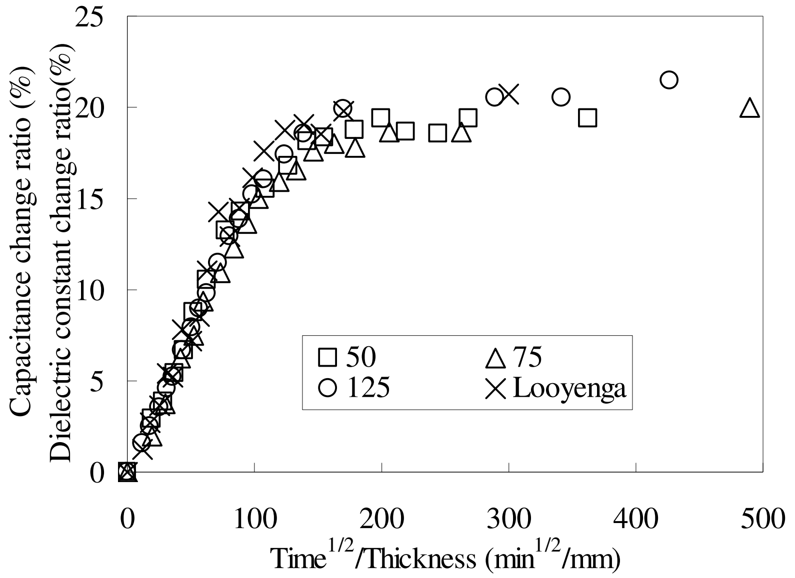


Fig. 6. Comparison between capacitance change of the sensor of several thickness films and estimate dielectric constant change calculate by using Looyenga equation (moisture absorption test)

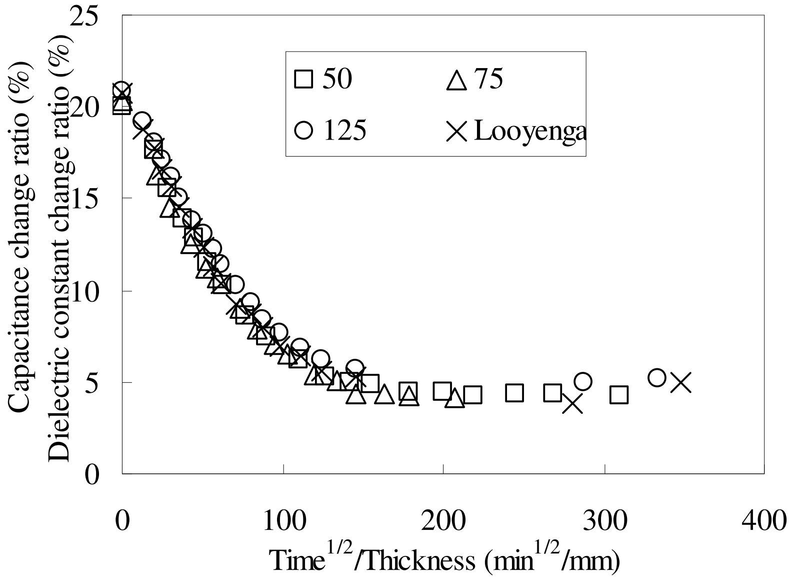


Fig. 7. Comparison between capacitance change of the sensor of several thickness films and estimate dielectric constant change calculate by using Looyenga equation (moisture desorption test)

Results and discussion. The capacitance changes of moisture absorption tests and desorption tests are shown in Fig.6 and Fig.7, respectively. The ordinates are the dielectric constant change ratio, which is equal to the capacitance change ratio, and the abscissa is $\sqrt{t}/d$ to normalize the influence of thickness. The results indicate that there is no influence of thickness from 50μm to 125μm. Looyenga's equation is accurate for the material used in this study to estimate the moisture absorption from the capacitance change of the sensor.

Experiment of sensor embedded in GFRP laminate

In order to demonstrate the feasibility of the sensor to measure the moisture absorption of FRP, the sensor is embedded in GFRP laminate, and internal moisture absorption ratio of GFRP laminate is measured.

Specimen configuration is shown in fig.8. A 125μm thickness sensor covered with a 7.5μm polyimide film to protect the electrode is inserted between 2 plies of 50×50(mm) woven GFRP prepreg, and cured at 130°C for 90 min by hotpress. The thickness of the specimen is 0.475mm. In order to compare the moisture absorption ratio of GFRP to the capacitance change of embedded sensor, moisture absorption test is carried out. After desiccation of the specimen, it is immersed in a water bath filled with purified water at 50ºC. Then, the moisture absorption ratio of the specimen and the capacitance change of the sensor are measured. Electronic Balances manufactured by Shimadzu Cooperation is used to measure the weight of GFRP, and 3532-50 LCR HiTESTER with 9262 test fixture manufactured by HIOKI E.E. Cooperation is used to measure the capacitance of the sensor at 1kHz. All the measurements are carried out after wiping surface water by paper filter.

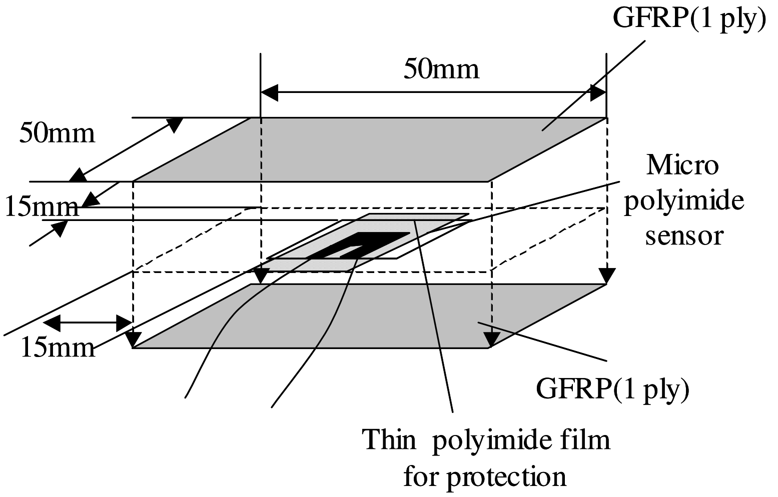


Fig. 8. Configuration of GFRP specimen with an embedded sensor

The moisture absorption ratio of FRP surrounding the sensor is estimated from the sensor output as follows. Assume that the volume fraction ratio of water in epoxy resin $v_{\mathrm{w\_epoxy}}$ is equal to that in polyimide $v_{\mathrm{w\_PI}}$. The volume fraction ratio of water in FRP $v_{\mathrm{w\_FRP}}$ can be calculated as follows.

$$v_{w\_FRP} = \frac{v_{w\_epoxy}\left(1-v_f\right)}{1-v_f v_{w\_epoxy}} = \frac{v_{w\_PI}\left(1-v_f\right)}{1-v_f v_{w\_PI}} \tag{8}$$

where v_{f} is the fiber volume fraction of FRP. The weight fraction ratio of water in FRP w_{w} is

$$w_w = \frac{v_{w\_FRP}\,\rho_w}{\rho_{FRP} + v_{w\_FRP}\left(\rho_w - \rho_{FRP}\right)} \tag{9}$$

where ρ_w is the density of water and ρ_{FRP} is the density of FRP. Moisture absorption ratio is calculated as follows.

$$M = \frac{w_w}{1 - w_w} \tag{10}$$

In this experiment, ρ_{FRP}=1633 kg/m^3, and v_f=0.5.

Results and discussion. Since the GFRP specimen is thicker than the sensor and the sensor is embedded in the midplane, it is supposed to take time to defuse water to the sensor. The moisture absorption ratios of the GFRP specimen are shown in fig.9. The ordinate is the moisture absorption ratio, and the abscissa is $t^{0.5}$. Open circles represent the average moisture absorption ratio of the specimen calculated from the experimental weight gain. Open squares represent estimated moisture absorption ratio of the specimen surrounding the sensor, i.e. the moisture absorption ratio of GFRP in the midplane. The average moisture absorption obeys Fickian low in the initial stage, and saturates with increase of time. On the other hand, there is a delay of the onset of the change for the estimated moisture absorption ratio in the midplane. This result is apparently different from the results of the sensor itself shown in Fig.3-5, and means diffusion of water in the laminate. The experiment clearly shows that the sensor has capability to measure the internal moisture absorption ratio.

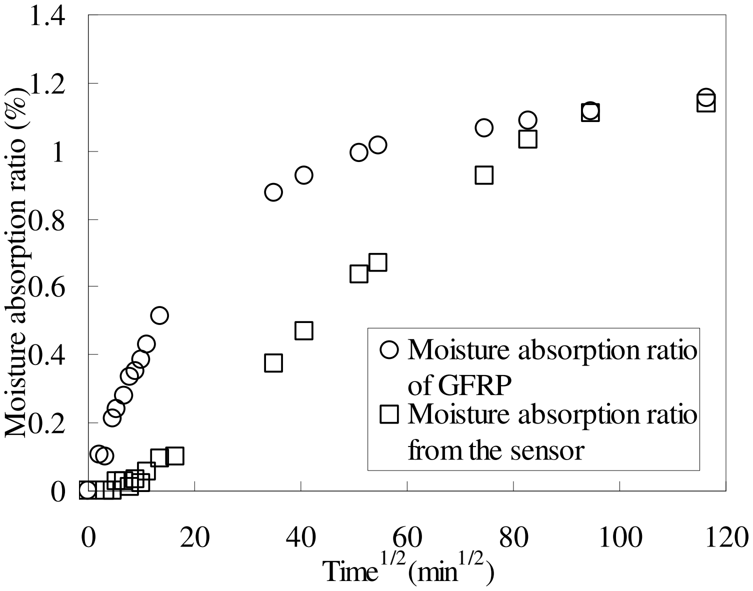


Fig. 9. Average moisture absorption ratio and that in the midplane of GFRP specimen

Concluding remarks

In this study, a new method to measure the moisture absorption ratio of FRP by the change of dielectric constant, which corresponds to the capacitance, of a micro polyimide sensor embedded in FRP is proposed. At first, the relationship between the moisture absorption ratio and the dielectric constant change of the sensor itself is investigated. Then, GFRP specimens in which the sensor is embedded in the midplane are immersed in water, and the moisture absorption ratio of the specimen and the dielectric constant change of the sensor are monitored. As a result, it is shown that moisture absorption ratio of GFRP can be easily measured by the proposed method.

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