# OPTICAL FIBER SENSOR TO MONITOR THE CHEMICAL DEGRADATION OF FRP

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**SUMMARY**: Polymeric materials such as FRP has been used in chemical plant under acidic or alkaline condition for anti corrosion materials. These materials also degradate under severe condition, then they show obvious change in chemical structures due to hydrolysis or other reactions. Sometimes chemical degradations result in fracture of chemical equipment. Therefore, the development of the on-line monitoring system for the condition of equipment is expected. In this study, possibility of new on-line monitoring system as smart materials was examined. In order to detect the chemical changes of surroundings, two types of monitoring methods with the optical fiber, were investigated. The results showed that it is possible to detect the chemical degradation of FRP by these methods.

**KEYWORDS**: Optical fiber sensor, Smart structure, Hydrolysis, Infra-red light, Infra-red Spectroscopy, On-line monitoring, Corrosion, FRP

## 1. INTRODUCTION

Recently, optical fiber sensors have been noticed for sensing material in the smart structure, and many studies have been reported [1, 2]. Then it can be used for many applications. Today, a fine fiber embedded in the matrix as the sensor, has been developed to get information about internal strain, temperature or mechanical damage of composite. The process of inspection and maintenance of the equipment with these smart materials will be much different from usual manner because these materials make it possible to monitor the condition of equipment with on-line.

We have been studying the corrosion behavior of polymeric materials such as FRP used in chemical plants [3~5]. As a result of the experiments, plastics exposed under acidic or alkaline condition showed obvious change in chemical structures due to hydrolysis or other reactions. This change can be easily analyzed by using an infra-red spectrometer (IR). Therefore, it is expected that the on-line monitoring system for the condition of equipment will be developed.

In this study, we examined the possibility of new system with optical fibers which are embedded in a material for monitoring chemical structures in the composite. Two types of sensors used for the monitoring experiment were based on refractive index monitoring and evanescent wave spectroscopy.

## 2. EXPERIMENTAL

### 2.1 Refractive Index Monitoring

Optical fiber refractive index sensors were prepared from plastic optical fiber (POF). POF doesn't give error signal by electromagnetical factor and has plastic characteristics such as good performances in light weight, deformability and workability comparing with glass optical fiber. A 50mm length of clad was stripped at the center of a 2m long fiber. The ends of fiber were connected to light source and detector of spectrophotometer. The intensity of light at the detector depends on the refractive index at the stripped region. Therefore, if the stripped region is placed in a sample, the change in refractive index of the sample results in a change in intensity of light at the detector. This technique is not affected by wavelength of light, since the refractive index will change at all wavelengths.

### 2.2 Evanescent Wave Spectroscopy

The chalcogenide glass fiber which can transmit infra-red spectral region (wave length of 3μm ~ 8μm) was used as second type optical fiber sensor in this study. The sensor was prepared by stripping the clad from the middle part of fiber which is approximately 30mm length. When light undergoes total internal reflection in the core of an optical fiber, light don't reflect exactly at the core / clad boundary but penetrate into the cladding slightly then reflect there. This region where light penetrate into is known as the evanescent field. Transmitted infra-red light gives the information of sample contacted to the exposed fiber core at the evanescent field. Therefore, the spectrum shows a character of the sample in this wavelength. A module with 1-35 fibers as shown in Fig.1 was made for this study. This module was put in the sample room of FT-IR.

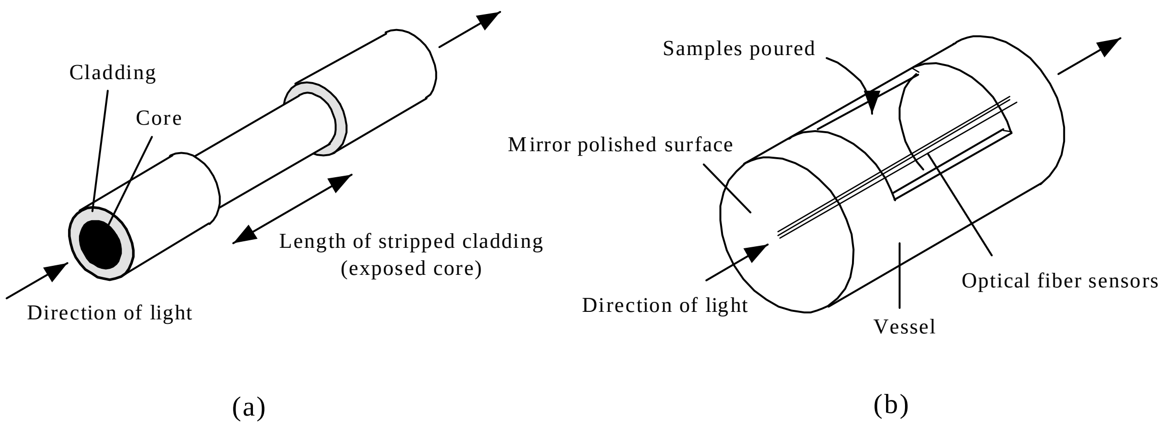


Fig.1 Schematic diagrams of (a) structure of one optical fiber, (b) module with IR optical fiber sensors.

A visible-light transmittable glass fiber was also embedded in bundle of the chalcogenide fibers, and the optical axis was adjusted with laser light irradiated through the visible-light optical fiber. Before measuring of spectra of samples, background spectra were collected using an empty module. In this study, 100 times repeated spectra from 800 ~ 4000cm$^{-1}$ were collected in order to improve signal-to-noise ratio.

## 3. RESULTS AND DISCUSSION

### 3.1 Refractive Index Monitoring

At first, in order to check if the POF sensor can detect the change of refractive index, the absorbance of POF was monitored with change the concentration of solution around the sensor. Glycerine solution was used as sample for this monitoring check, because refractive index of glycerine solution depends on concentration considerably. The results showed that the absorbance of POF decreased as decreasing of the concentration of solution. Fig.2 shows the change of absorbance with refractive indices that corresponded with concentration of glycerine solution. It can be seen from this figure that absorbance increased when refractive index of sample approached refractive index of core. It is confirmed that POF sensor can detect the change of refractive index around the sensor by monitoring absorbance.

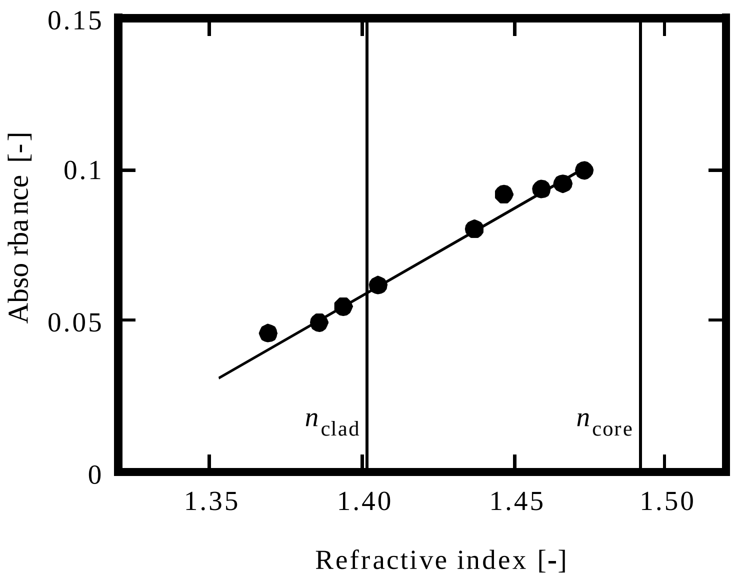


Fig.2 Effect of refractive index on absorbance.

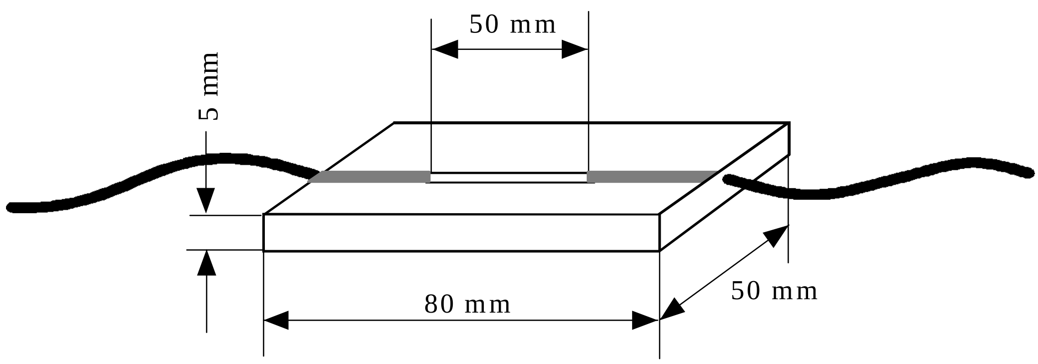


Fig.3 Illustration of specimen for POF monitoring test.

The POF sensor was embedded in isophthalic acid type unsaturated polyester resin, which have been used for the matrix of corrosion resistant FRP. Fig.3 shows size and shape of a specimen used for POF monitoring test. This specimen was immersed in 30wt.% sodium hydroxide solution at 80 . The absorbance of POF was monitored while resin was degradated. Fig.4 shows the change in absorbance of POF obtained from twice experiments. The initial value of absorbance was still constant and suddenly increased more than ten hours after.

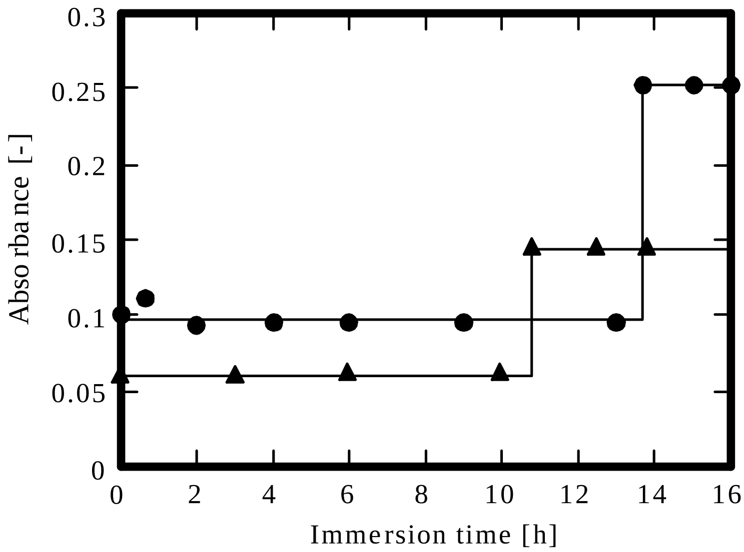


Fig.4 Change of absorbance in POF monitoring test.

Fig.5 shows photograph of cross section of specimen after POF monitoring experiment. The boundary between the outside discolored layer and the central unchanged layer was clearly observed. The result in IR-analysis of both layers showed that ester groups were cut by hydrolysis reaction in the discolored layer. For example, spectrum of discolored layer differed with that of unchanged layer in a peak near 1700cm$^{-1}$. Then, this outside layer was corroded layer.

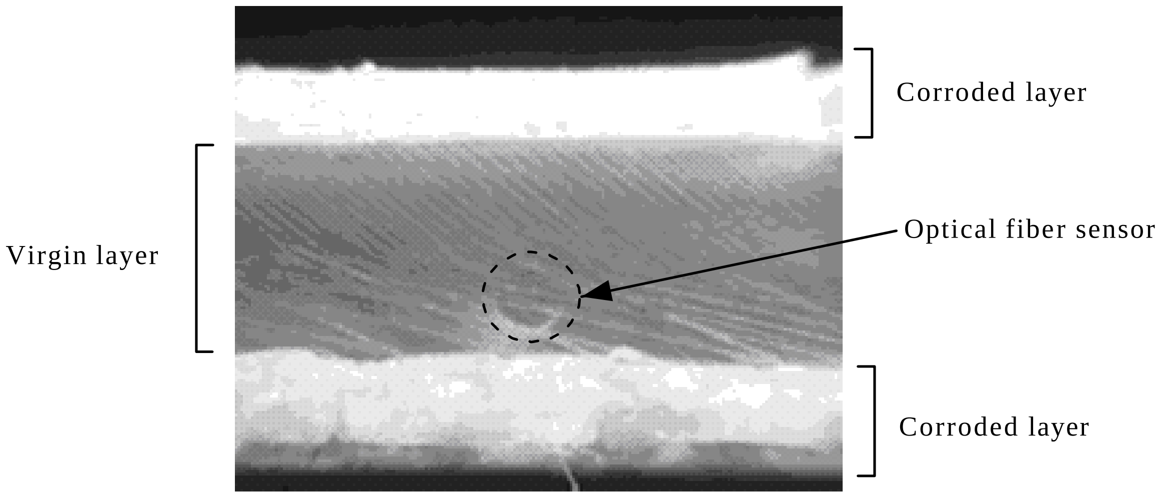


Fig.5 Observation of cross section of specimen after POF monitoring experiment.

Then, specimen cross section was analyzed using SEM and EDS, however, the results showed that the corroded layer didn't reach the POF sensor. Also, the element of Na was not detected around the sensor in EDS-analysis. However, the sensor detected some change because this experiment has good reproducibility.

### 3.2 Evanescent Wave Spectroscopy

At first, in order to obtain the basic knowledge for preparing modules, experiment was carried out using module with various number of fibers. Fig.6 shows the change in transmission energy of IR obtained from some modules which has different number of fibers. From Fig.6 it can be seen that transmission energy increased with number of fibers.

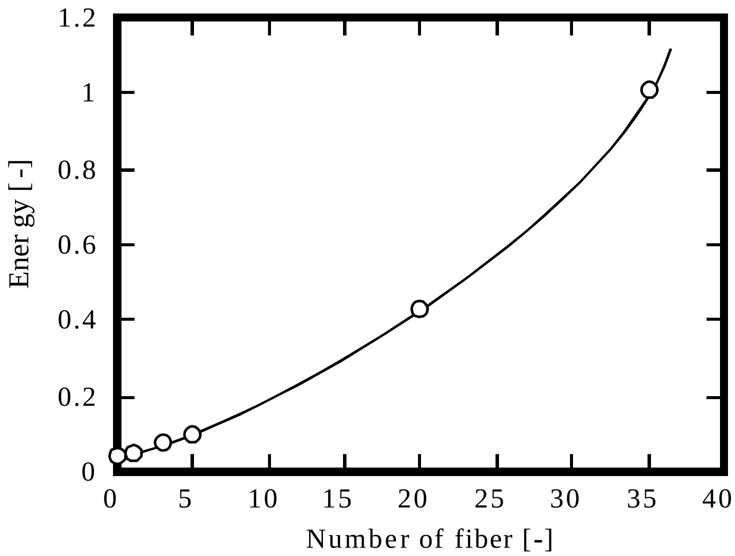


Fig.6 Change in transmission energy of IR obtained from optical fiber sensor.

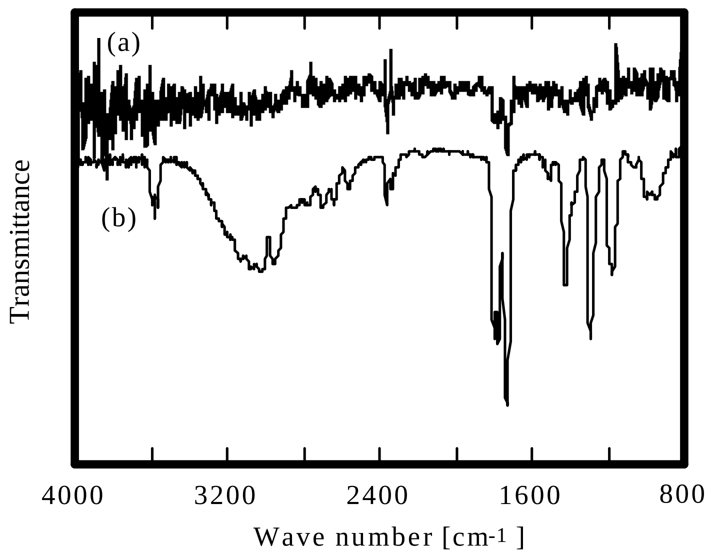


Fig.7 IR spectra of acetic acid obtained from IR optical fiber method using (a)5 fibers, (b) 20 fibers.

Then, acetic acid solution which shows a characteristic IR spectrum at near 1700cm$^{-1}$ was introduced into the module as a sample, and the change of spectra with the number of fibers was observed. Fig.7 shows examples of spectra obtained from this measurement using 5 and 20 fibers. The results showed that the energy transmitted by the module decreased with decreasing the number of fibers, and five fibers were the minimum number to measure spectra in this method.

In order to check that the module can detect the spectra of other liquid samples, ethanol and mixed solution of ethanol and acetic acid were added to the module respectively. Fig.8 shows spectra of three solutions obtained by 20 chalcogenide fibers module.

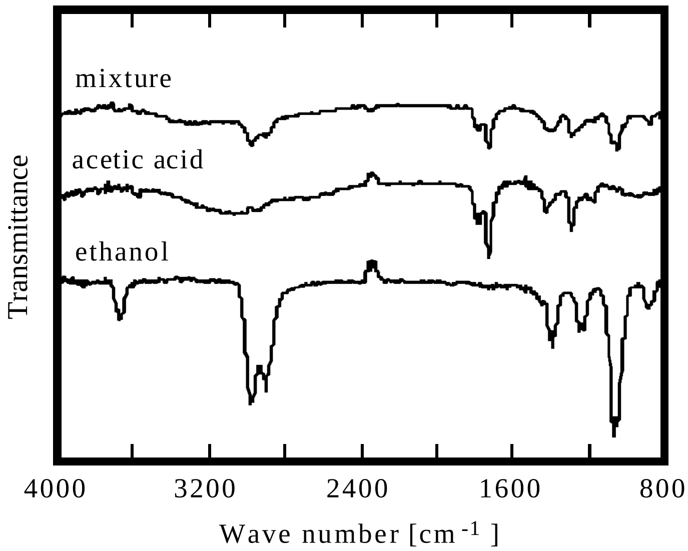


Fig.8 IR spectra of various solutions obtained from 20 fibers module.

Both acetic acid and ethanol spectra were almost equivalent to them obtained by general method. In addition, the spectrum of mixture was a summation of spectra of two solutions. It is concluded that chalcogenide fiber sensor can be sensed chemical structures of liquid samples. The system can be applied to monitor the degradation of resin by corrosion.

## 4. CONCLUSIONS

In this study, we examined the possibility of new system with optical fibers which are embedded in a material for monitoring chemical structures in the composite. Two types of sensors used for the monitoring experiment were based on refractive index monitoring and evanescent wave spectroscopy.

In experiments of refractive index monitoring, POF sensor can detect the change of refractive index around the sensor by monitoring transmittance. It appeared that there is the possibility of optical fiber sensors monitoring the chemical degradation of unsaturated polyester resin.

In experiments of evanescent wave spectroscopy, it showed that chalcogenide fiber can sense the chemical structure of sample contacted with stripped core as IR spectra. It is assumed that chalcogenide fiber can monitor the chemical degradation in FRP by detecting the change of the chemical structure in resin before and after corrosion. Chalcogenide fiber sensor is expected its applications because this sensor can detect not only penetration of environmental solution into resin but also change of chemical structure caused by hydrolysis.

If smart materials investigated in this study will be developed, it is expected that maintenance cost will be lower and the reliability of equipment will be improved.

## 5. ACKNOWLEDGEMENTS

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