Development of Acoustic Emission and Gas Monitoring Methods for Nondestructive Detection of Termite Attack on Wooden Structures

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Contents

Introduction

At present, the damage by two subterranean termites, *Coptotermes formosanus* and *Reticulitermes speratus*, to the wood and wood-based materials in the wooden constructions was severe in Japan. The damage by a drywood termite *Incisitermes minor* has been also extending in Japan (Yanase *et al*. 2001, 2012; Indrayani *et al*. 2005).

Increased attention has recently been paid to wood preservation using little or no chemicals, which would be beneficial for both human health and the environment. Many methods and materials have been developed, involving less harmful chemicals, physical barriers using stainless-steel mesh or particulate materials (Ebeling and Pence 1957; Su *et al*. 1991, 1992; Tamashiro *et al*. 1991; Sornnuwat *et al*. 1995; Yanase *et al*. 2005, 2009), and microwave treatment (Lewis *et al*. 2000; Nakai *et al*. 2009). However, they do not always work perfectly. For example, while less harmful chemicals are less toxic to humans and more environmentally friendly, they are also less toxic for termites, and thus their long term performance can not be guaranteed. The most realistic solution for obtaining ideal wood preservation is the combination of two processes: precise evaluation of bio-deterioration in wood and treatment using a minimum amount of chemicals.

At present, termite attacks, which will be taken to mean the damage of wood by the worker termite feedings in this study, are almost always detected by visual inspection, but it is difficult to detect the early stages of termite attack by visual inspection alone. Currently, nondestructive methods of inspecting termite attack are not widely used in Japan. In the future, however, efficacious and nondestructive detection methods will be required to assist in termite control to reduce the use of termiticide or to implement a chemical-free management scheme.

Several methods for nondestructive detection of termite attacks have been investigated and developed. They are classified into two categories: evaluation of mass loss of wooden objects and detection of termite activity in wooden constructions. One of the former methods is the detection of inner cavities by measuring the velocity

or attenuation of the ultrasound or elastic wave propagating in wood (Mori *et al*. 2010, 2011), and by using X-ray computer tomography (Fuchs *et al*. 2004). A scanning radar apparatus at 1.4 GHz, which detects the reflection of the wave radiated onto the wood, was applied for the nondestructive detection of an inner defect in wood such as a cavity (Fujii *et al*. 2000, 2006; Fujii and Yanase 2001).

An example of the latter is acoustic emission (AE) monitoring, which utilizes the detection of small elastic waves called AEs. When wood is attacked by termites, micro-fractures are generated in the wood by the feeding of worker termites. As a result, elastic waves, or AEs, are generated due to the release of the strain energy stored in the wood (Fujii 1997a). AEs could be detected in wood specimens under termite attack, and the results obtained in their laboratory- and field-tests suggest that AE monitoring may be an effective non-destructive method for detecting the presence and activity of termites in wood, even if the attack is still in its incipient stage (Fujii *et al*. 1989, 1990a, 1990b; Noguchi *et al*. 1991; Imamura *et al*. 1991). The feeding behavior of termites was also observed by using a microscope and CCD camera to better understand its relation to the generation of AEs (Fujii *et al*. 1995; Matsuoka *et al*. 1996; Indrayani *et al*. 2004, 2007). They reported that the amplitudes of the AEs generated by termite attack depended on the type of feeding by the worker termites.

AE monitoring has been applied for the detection of termite attack in the Japanese wooden constructions, and the locations where termites attacked wooden members were detected and treated by a minimum amount of chemicals (Fujii *et al*. 1998; Yanase *et al*. 1999). AE detector has also used for locating the feedings of termites in wooden construction when installing and inspecting aboveground termite bait station (Weissing and Thoms 1999), for detection of termite infestations in the urban trees with a much high moisture content (Mankin *et al*. 2002), and for monitoring the infestation of termites to the wood stakes as trapping around an electrical power plant (Fujii 1997b). AE monitoring was also applied for the evaluation of an effect of the chemical treatment on termite infestations (Thoms 2000; Lewis *et al*. 2004).

Lewis *et al*. (1991) and Lemaster *et al*. (1997) noted that the sensitivity of an AE transducer depended on the resonant frequency of AE transducers and concluded that

more detailed studies of the optimal measuring system were needed if AE monitoring was to be used as a practical non-destructive method for detecting termites. They also pointed out the need for a waveguide, such as a lag screw attached to the AE transducer that penetrated walls to reach the inner posts or studs, to effectively detect AEs generated inside of house walls.

Feasibility of method to detect reflection of ultrasound wave from the movement of cottonwood borer of *Plectrodera scalator*, not termite, in the wood packing materials has been investigated (Fleming *et al*. 2005). Technology to detect reflection of micro (Evans 2002, Indrayani *et al*. 2006) or millimeter (Fujii *et al*. 2007) waves from termite movement in the wood has also been developed and applied. The method to monitor the interruption of an electric current (Su *et al*. 2000; Su 2001, 2002) and an infrared light (Pallaske *et al*. 2001) by termite activity were also developed for application to bait station.

Detection of odors from termite colonies with the help of trained dogs could be a possible solution. The abilities of the dogs to detect varying numbers of subterranean termites, differentiate between five termite species, discriminate termites from termite-damaged wood, and distinguish cockroaches and carpenter ants from subterranean termites have been investigated (Brooks *et al*. 2003). Trained dogs were 96% accurate in finding over 40 termite workers and incorrectly indicated the presence of termites in only 2.7% of the containers without termites. The ability of trained beagles and electronic odor sensing devices to detect gases given off by termites has been also investigated (Lewis *et al*. 1997). In the experiments using wooden blocks with different densities of subterranean termites, the percentage of blocks correctly identified by beagles was reportedly 81%. This was higher than that obtained using electronic odor sensing devices (48% accuracy) that primarily detect methane gas. These findings demonstrate the ability of trained dogs to detect termites, although the cost and time employed in training the dogs are not always practical.

It was also reported that the most common and abundant metabolic gases emitted from termite colonies were carbon dioxide (CO_2) and methane (CH_4), and other gases such as chloroform ($CHCl_3$), nitrous oxide (N_2O), carbon monoxide (CO), and

hydrogen (H_2) might also be emitted (Zimmerman *et al*. 1982; Khalil *et al*. 1990; Sanderson 1996; Sugimoto *et al*. 1998; Ohkuma 2003). There is a possibility that the detection of these gases provides a nondestructive method to detect early termite attack. Recently, semiconductor gas sensors with high sensitivity to detect odor, methane, and hydrogen were developed (Suzuki and Takada 1995; Katsuki and Fukui 1999), and these sensors can possibly detect termite attacks with the high accuracy and efficiency.

As stated above, a lot of methods have been developed for nondestructive detection of termite attack on wood. For their practical application, however, several problems still remain to be solved. For examples, it is difficult to detect termite attack on wood structures with a limited number of AE transducers, and it is necessary to improve a gas sensor for efficient detection of termite activity.

This thesis consists of two Parts, containing five Chapters. Part I (Chapters 1 and 2) focuses on the feasibility of polyvinylidene fluoride (PVDF) film and steel waveguides for the detection of AEs generated by termite attack in wood. Part II (Chapters 3-5) discusses the application of semiconductor gas sensors to the detection of termite attack in the several conditions.

Chapter 1 discusses the feasibility of PVDF films as a new AE transducer in comparison with conventional lead zirconate titanate (PZT) elements. Chapter 2 discusses two waveguides, a metal plate attached to the wood surface and a metal needle penetrated to inner posts or studs, and their feasibility for AE detection of termite attack. Chapter 3 deals with three types of semiconductor gas sensors (odor, methane, and hydrogen sensors) for the detection of metabolic gases emitted by termites. Chapter 4 discusses a gas detection apparatus equipped with a semiconductor gas sensor and its feasibility for qualitative and quantitative measurement of hydrogen and methane emitted by termite feedings under several conditions. Chapter 5 discusses a long term measurement of hydrogen and methane emitted by termites, relationships between AE and gas concentrations, and the effect of ambient relative humidity on concentrations of hydrogen and methane emitted by termites under a ventilation system.

Part I

Novel Methods for Detection of AE Generated by Feeding of Termites

Chapter 1

Use of Polyvinylidene Fluoride (PVDF) Film as an AE Transducer

1.1 Introduction

An element of lead zirconate titanate (PZT) is one of the most frequently used piezoelectric materials for AE transducers with high sensitivity (Hamstad 1995). However, it is inflexible and relatively expensive. New materials to overcome such shortcomings are expected for practical application to detect AE generated in the wood and wood-based materials. Polyvinylidene fluoride (PVDF), which has a repeat unit of CH_2-CF_2, is a piezoelectric polymer, and shows the highest piezoelectricity among these materials (Nakamura *et al*. 1983). Although it is less sensitive than the PZT element, it is provided as thin and flexible membrane, able to cover a much larger area, suitable for mass-production, and more cost-effective (Yanase *et al*. 2000). The thickness of the membrane has also a great influence on its sensitivity, and the thicker the membrane, the higher the sensitivity. Thus, it may be reasonable to use PVDF as an AE transducer by attaching it to wood surfaces or by inserting it among the construction members of the wooden buildings (Yanase *et al*. 1998). When a film with a broad area or a multi layered film in used, the resultant sensitivity for detecting AE wave should increase.

In this chapter, the feasibility of PVDF film as an AE transducer was examined for the detection of AE wave generated by termite attack in the wood and wood-based materials used in the wooden constructions.

In this chapter, the terms of "PVDF sensor" and "PZT sensor" will be taken to mean AE transducers consisting of PVDF film and PZT element, respectively.

1.2 Materials and methods

1.2.1 Detection of artificial AEs using PVDF sensor (Experiment 1)

Some typical properties of PVDF film (Fig. 1.1) and its comparison with some other piezoelectric materials are shown in Table 1.1. PVDF has the highest piezoelectric constant in g_{31} among all the materials listed, which means it has the highest piezoelectric sensitivity, while PZT (Fig. 1.1) has the highest piezoelectric constant in d_{31}. PVDF film has also a wide frequency response range, which makes suitable for AE transducer (Nakamura *et al*. 1983). PVDF film is flexible other than the ceramic piezoelectric element. In addition the density is much less resulting in significantly better acoustic impedance matched to wood. However, the relative dielectric constant of PZT is much higher. The piezoelectric stress constant for PZT is less than one tenth of that for PVDF. Hence, for a given stress more output from PVDF sensor would be obtained (Hamstad 1995).

PVDF film (40 μm thick, KF Piezofilm, Kureha Corporation) is uniaxially stretched before polling and the greatest piezo-electric effect appears in this direction. Both surfaces of the PVDF film are coated with aluminum under vacuum evaporation to form electric terminals. In this study, a low-noise cable was attached to the terminals of the PVDF film using a conducting paste, and the PVDF film was grounded to reduce noise. The signal from the film was transferred through the cable to an amplifier.

Sugi (*Cryptomeria japonica*) was cut into a specimen 570 mm long with a cross-section of 310 by 45 mm, and air-dried. PVDF films (PVDF sensors) of 15, 20, 25, and 40 mm square were attached to the radial section of the specimen with adhesive (Fig. 1.1). A cylindrical piezoelectric transducer (PZT sensor with 22 mm high, 8 mm in diameter, AE-901U, NF Corporation) with a fundamental resonant frequency of 70 kHz or 100 kHz was also attached to the specimen using a rubber band. Silicone grease was used as an acoustic coupler. To investigate the ability of PVDF sensors to detect AEs propagating in wood, the signal generated by breaking a

pencil lead by pushing it on the specimen surface was used as an artificial AE source. A pencil lead of 0.5 mm in diameter was broken into 3 mm lengths. This method can cause a momentary release of load which exhibits a step function and includes the frequencies required for AE measurement in a reproducible manner (Nakao *et al* 1986; Okumura *et al* 1988). An artificial AE wave was generated at a distance of 150 mm from the center of the sensor and propagated longitudinally or radially, as shown in Fig. 1.2. Signals from PVDF and PZT sensors were amplified by 40 dB and discriminated at a threshold voltage of 0.2 V. Neither highpass nor lowpass filters were used. The frequency spectra were analyzed with a FFT analyzer (CF-5220, ONO SOKKI Co., Ltd.).

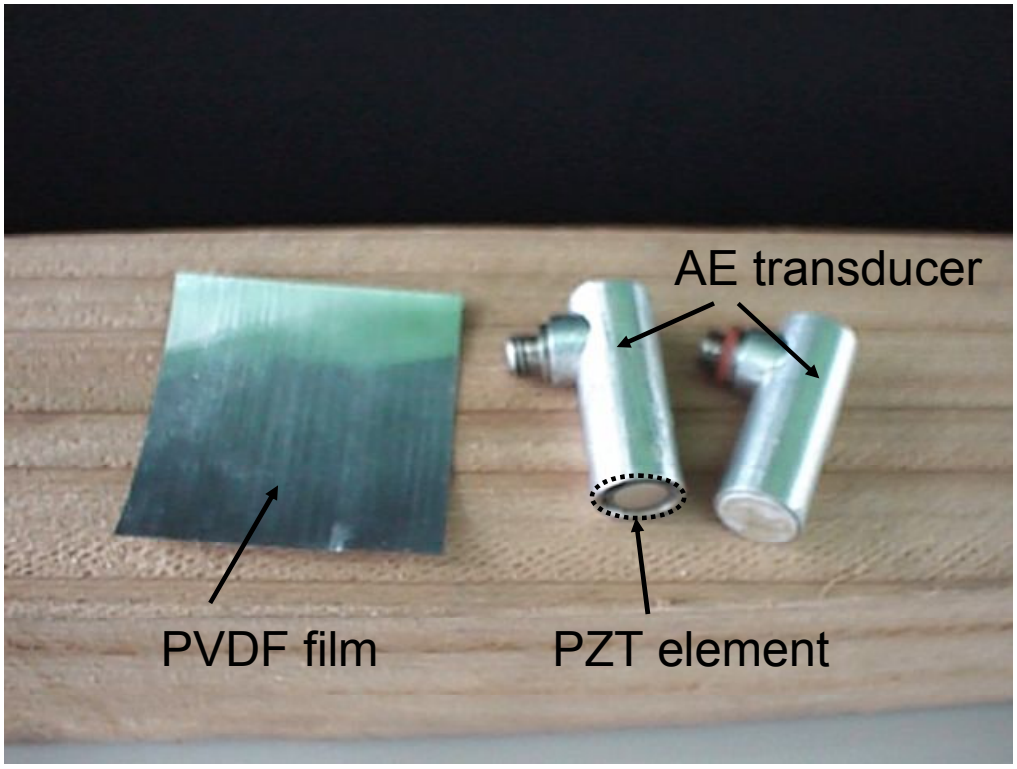

Fig. 1.1. PVDF film and AE transducer (PZT element).

Table. 1.1. Properties of some piezoelectric materials (Wang *et al*. 1998).

Piezoelectric constants \ Material	Polyvinylidene fluoride (PVDF)	Polyvinyl fluoride (PVF)	Lead Zirconate titanate (PZT)	Barium titanate ($BaTiO_3$)	Crystal
Dielectric constant ε	13	5	1200	1700	4.5
Piezoelectric strain constant d_{31} (10^{-12} C/N)	20	1	110	78	2
Piezoelectric stress constant g_{31} (10^{-3} V·m/N)	174	20	11	5	50
Electromechanical coupling coefficient k_{33}	0.10	—	0.31	0.21	0.09
Density ρ (10^3 g/cm^2)	1.78	1.38	7.5	5.7	2.65
Acoustic impedance Z(10^6 kg/m^2)	2.7	—	25	25	15.1

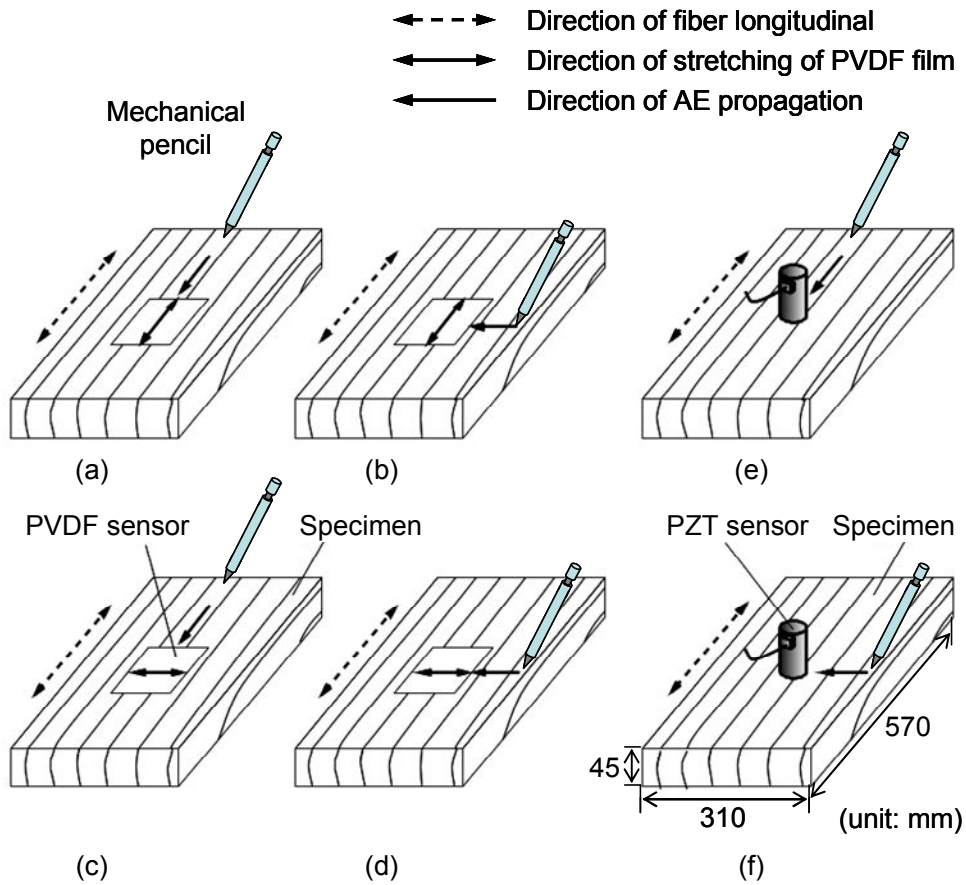

Fig. 1.2. Conditions for measuring the artificial AEs by a PVDF sensor ((a) to (d)) and a PZT sensor ((e) and (f)).

1.2.2 Attenuation measurement for artificial AE waves in wood and wood-based materials (Experiment 2)

Western hemlock (*Tsuga heterophylla*), Japanese red pine (*Pinus densiflora*), Japanese cedar (*C. japonica*) were cut into specimens of 500mm long with a cross-section of 50 by 50 mm, and air-dried. A five-ply plywood and a particleboard with 20 mm thickness and were also cut into specimens of 500 mm long and 50 mm width, and air-dried.

A PVDF sensor of 27 mm in length, 13 mm in width, and 28μm in thickness (SDT1-028K, Tokyo Sensor Co., Ltd) was attached to the surface of the wood specimen with double-faced adhesive tape. An artificial AE wave was generated on the surface by breaking a pencil lead of 0.5 mm in diameter and set in a mechanical pencil, where the lead was broken into a length of 3 mm. Signals from PVDF sensor was amplified by 85 dB, filtered using a high-pass filter of 50 kHz, discriminated at a threshold level of 0.6 V and recognized as an AE event.

1.2.3 Detection of AEs generated by termite attack using PVDF sensor (Experiment 3)

Western hemlock (*Tsuga heterophylla*) was cut into a small specimen 30 mm long (longitudinal) with a cross-section of 30 by 30 mm. A hole with 10 mm in diameter and 15 mm deep was drilled in the specimen and the specimen was allowed to air dry. A group of 100 worker termites of *Coptotermes formosanus*, which were collected from a colony maintained by the Research Institute for Sustainable Humanosphere, Kyoto University, was put into the hole and the opening was sealed with a silicon plug. PVDF sensors (single- and triple-layered; 25 by 25 mm) and a PZT sensor with a resonant frequency of 70 kHz were attached to the surface of the specimen with adhesive and silicone grease, respectively. Signals from the PVDF sensors were amplified 80 dB and discriminated at a threshold voltage of 0.2 V, while those from the PZT sensor 70 dB and 0.12 V. Another specimen of western hemlock (600 mm long; cross-section of 55 by 45 mm, with a hole of 10 mm in diameter drilled 30 mm

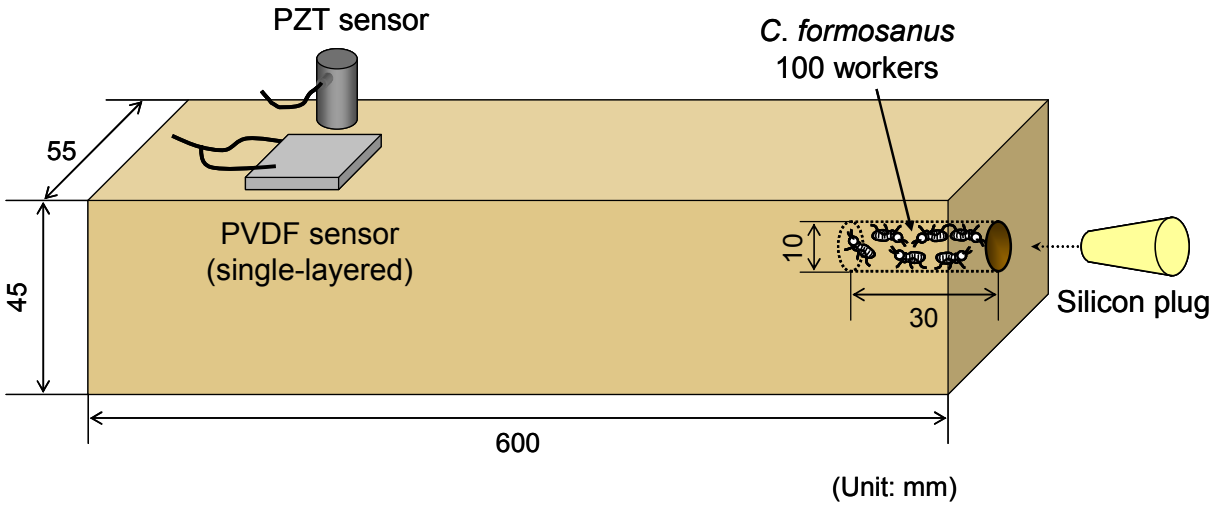

Fig. 1.3. Experimental setup for measurement of AE generated from termite feeding.

deep) was also prepared and allowed to air dry. A group of 100 worker termites of *C. formosanus* was placed into the hole (Fig. 1.3). The AEs generated by feeding of termites were detected by the PVDF sensor and the PZT sensor with a resonant frequency of 70 kHz, which were moved at several distances with 100 mm increments from the hole. Another PZT sensor was also attached just next to the hole as a reference sensor.

1.3 Results and discussion

1.3.1 Characteristics of AE detected by PVDF sensors (Experiment 1)

Figure 1.4 shows examples of the waveform and frequency spectrum of AEs detected by a PVDF sensor and a PZT sensor, after being amplified and discriminated. In both frequency spectra, a few peaks were observed under 10 kHz, which probably correspond to the natural frequencies of the longitudinal vibration of the specimen. There are some peaks near 70 kHz, the resonant frequency of the PZT sensor, are

observed in the spectrum for the PZT sensor. On the other hand, in the spectrum for the PVDF sensor, there is no discrete peak over 10 kHz, since the PVDF sensor has a lower Q factor

Figure 1.5 shows the average amplitudes of ten AE signals detected by PVDF sensors of different sizes, where the amplitude was estimated from the overall level of each frequency spectrum. The amplitude of the AEs that propagated radially was greater than that propagated longitudinally. It was reported that the attenuation coefficients of ultrasonic waves increased with frequency (from 0.1 to 1.5 MHz) either for longitudinal or shear waves in wood, and the lowest attenuation was obtained in the longitudinal direction (Bucur *et al*. 1992, 1994). This is contradictory to this study. When the amplitude was estimated from the peak-to-peak values of the detected signals, it was larger for the longitudinal direction than for the radial direction. Incidentally, a larger PVDF sensor provided a larger amplitude as a whole.

Figure 1.6 shows the average amplitudes of AE signals detected by four PVDF sensors in comparison with those detected by two PZT sensors. The average amplitudes for the PVDF sensors were about one tenth to one twentieth of those for the PZT sensors. A transducer consisting of three layers of PVDF film was on trial used to increase the amplitude of the PVDF sensor. The amplitude for this PVDF sensor was about twice that for a single-layered PVDF sensor. In addition, it was difficult to detect AEs using PVDF sensors of more than three layers because the background noise level increased and consequently the signal/noise ratio (S/N ratio) decreased.

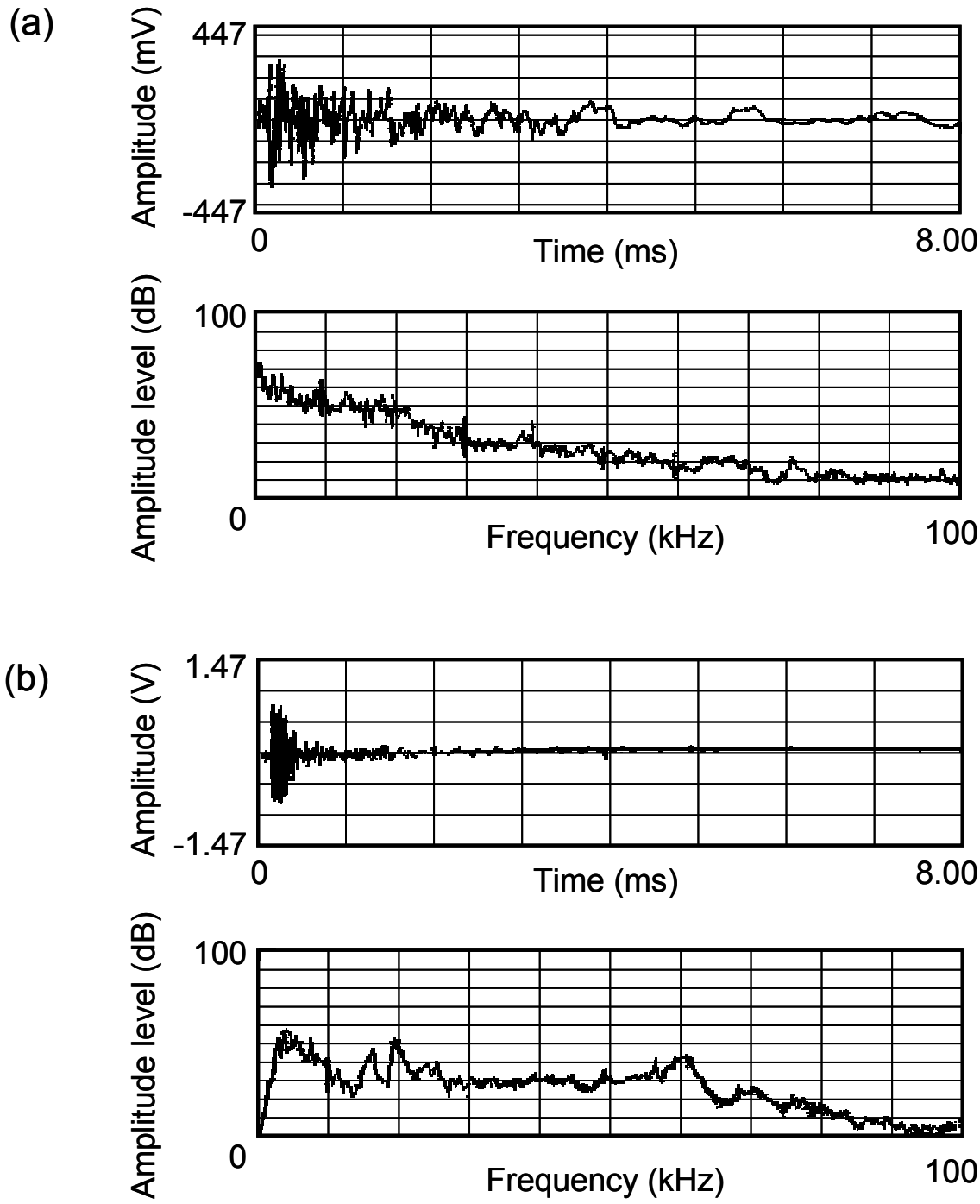

Fig. 1.4. Examples of the waveform and frequency spectrum of AEs detected by (a) a PVDF sensor and (b) a PZT sensor with resonant frequency of 70 kHz.

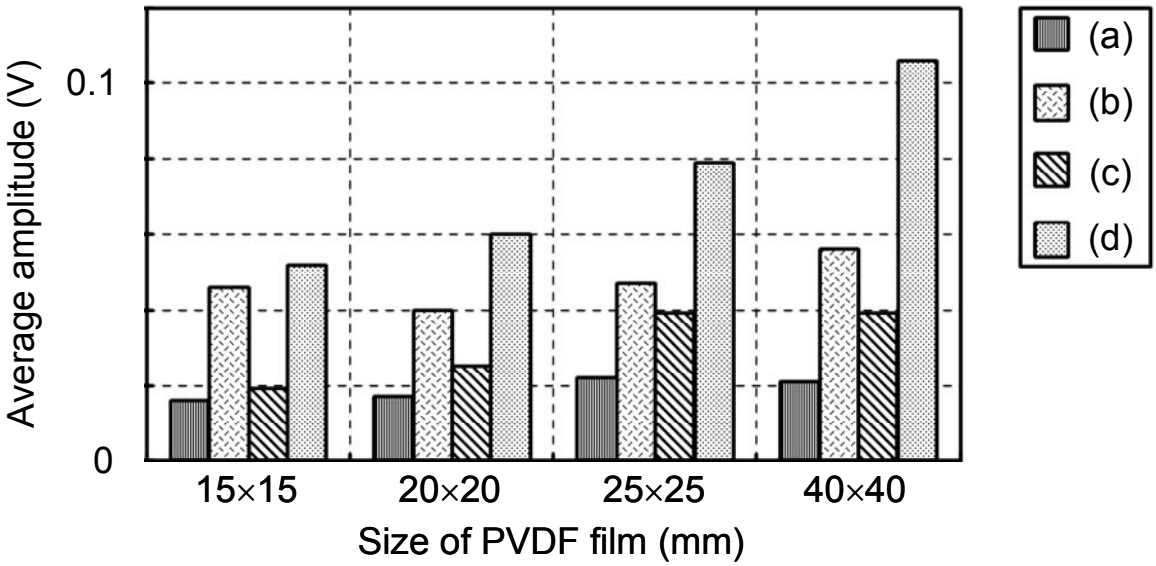

Fig. 1.5. Average amplitudes of artificial AEs detected by PVDF sensors.
(a) to (d): refer to Fig. 1.2.

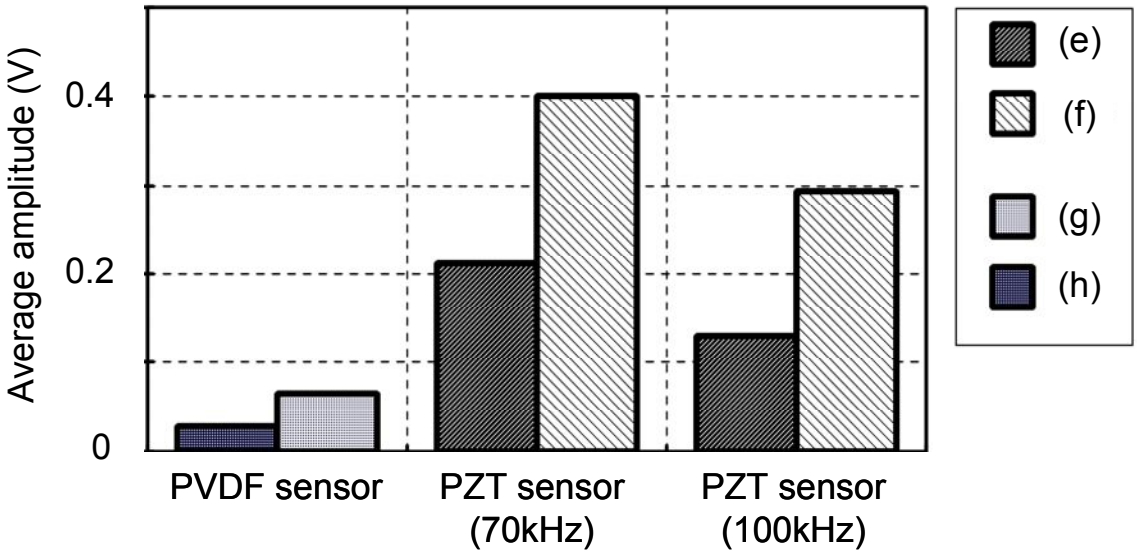

Fig. 1.6. Average amplitudes of artificial AEs detected by PVDF and PZT sensors.
(e) and (f): refer to Fig. 1.2. (g) and (h) correspond to the average amplitudes of all PVDF sensors of (a), (c) and (b), (d) in Fig. 1.4, respectively..

1.3.2 Attenuation of artificial AE wave in wood and wood-based materials (Experiment 2)

Figure 1.7 shows the relations of the peak-to-peak amplitudes detected by PVDF sensors to the distance between the center of PVDF sensor and artificial AE source. Artificial AEs were detected by PVDF sensors on the surface of specimens and it seemed that PVDF sensor could detect AEs generated within a propagation distance up to 450 mm on the wood and plywood specimens.

The amplitudes of AE waves propagated in the specimens, western hemlock, Japanese red pine, and Japanese cedar were larger than those in the plywood and the particleboard. In case of the particleboard, it was difficult for the PVDF sensor to detect artificial AEs over a distance of more than 250 mm. This implies that the artificial AE wave may be strongly attenuated at the boundaries between particle elements.

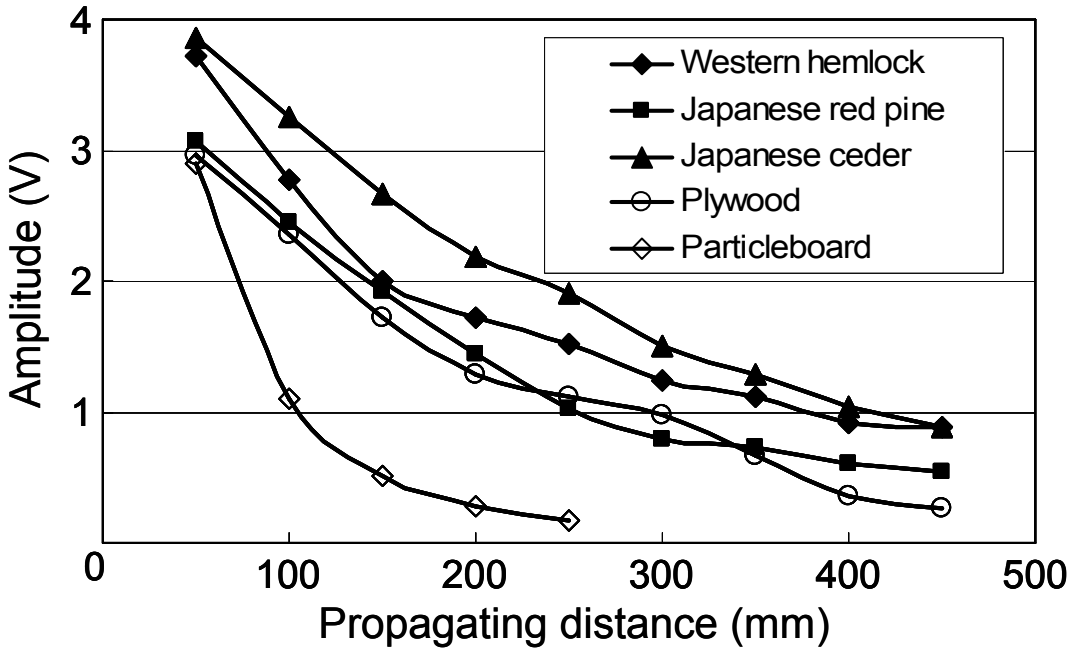

Fig. 1.7. Relations of the peak-to-peak amplitudes to propagating distance of artificial AE waves.

1.3.3 Detection of AEs generated by termite attack using PVDF sensor (Experiment 3)

Figure 1.8 shows the cumulative AE event counts detected by the PVDF sensors (single- and triple-layered) and the PZT sensor for specimens attacked by termites. The cumulative counts detected by the single-layered PVDF sensor were about one twelfth of those detected by the PZT sensor, while the cumulative counts detected by the triple-layered PVDF sensor were about one fifth of those detected by the PZT sensor. Figure 1.9 shows the relations of the relative cumulative AE event count to the distance from the AE source. The relative count decreased with distance from the AE source due to attenuation of the AE wave in wood and the attenuation in distance was more remarkable for the PZT sensor than for the PVDF sensor. This implies that PVDF sensor has sensitivity on the same level with PZT by layering three PVDF films and may be more profitable than PZT sensors to cover a wide area for monitoring the AEs generated by termite feeding in wood.

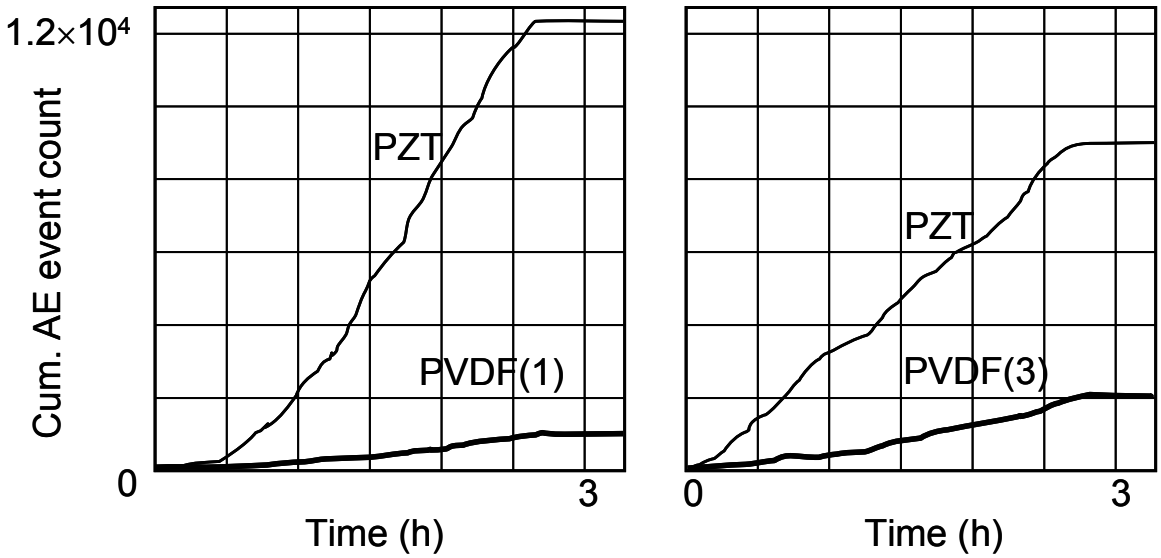

Fig. 1.8. Cumulative AE event counts with time for PVDF and PZT sensors.
PVDF (1): single-layered PVDF sensor, PVDF (3): triple-layered PVDF sensor.

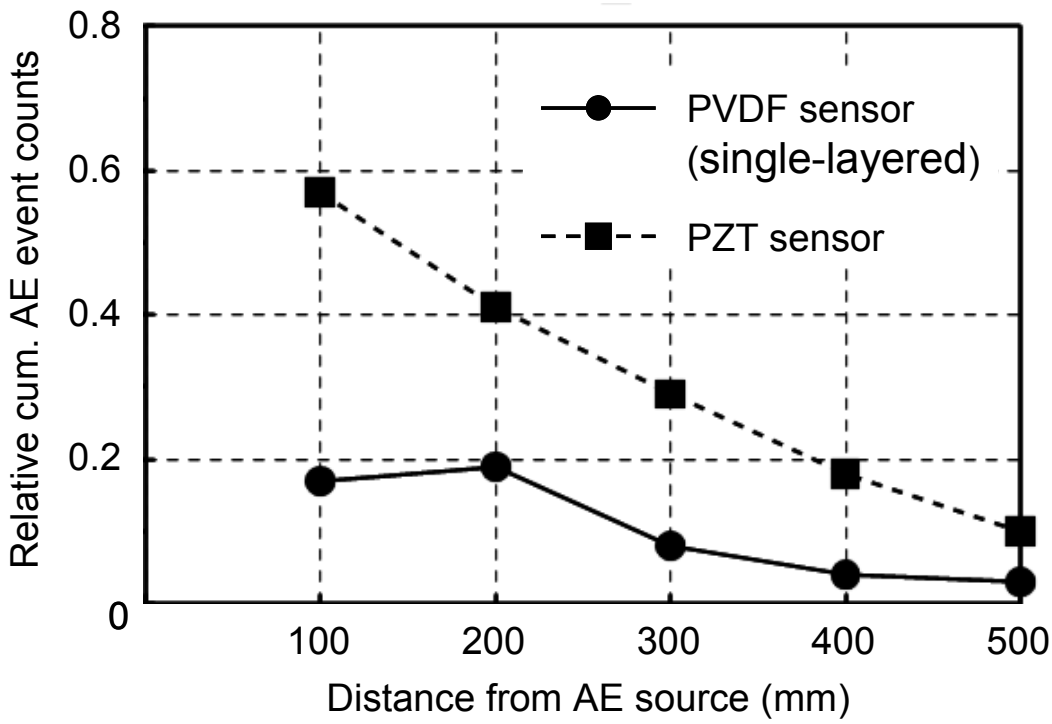

Fig. 1.9. Relationships between the relative cumulative AE event counts on the basis of the count at the reference PZT sensor and the distance from the AE source.

1.4 Summary

The feasibility of PVDF film with 40 μm thick as a new AE transducer was examined through detection of AEs from artificial AE source and feeding of termites to detect AEs effectively in the wood and wood-based materials used in the wooden constructions.

Three experiments were conducted as follows: artificial AEs generated by breaking a pencil lead was detected by PVDF film with five film sizes and PZT sensor (Experiment 1); artificial AEs were detected by PVDF sensor attached on the surfaces of three wood specimens and two wood-based materials at several distances with 50 mm increments from an artificial AE source (Experiment 2); AEs generated by feeding of *C. formosanus* were detected by single- and triple-layered PVDF sensors

attached on the surface of wood specimen at several distances with 100 mm increments from AE source for evaluating the detectivity of PVDF sensor (Experiment 3).

Although the sensitivity of PVDF sensor was lower than that of a PZT sensor, the sensitivity of a PVDF sensor could be increased by using multiple sheets (Experiment 1). It was difficult for the PVDF sensor to detect artificial AE coming over the distance more than 250 mm in the particleboard among three wood specimens and two wood-based materials, because the artificial AE wave was strongly attenuated at the boundaries between elements such as wood particles (Experiment 2). For the detection of AEs generated by termite attack, the cumulative counts detected by the single-layered PVDF sensor were about one twelfth of those detected by the PZT sensor, while the cumulative counts detected by the triple-layered PVDF sensor were about one fifth of those detected by the PZT sensor (Experiment 3). PVDF sensor may be more profitable than PZT sensors to cover a wide area for monitoring the AEs generated by termite feeding in wood.

Chapter 2

Steel Waveguides for Detecting AEs Generated by Termite Attack

2.1 Introduction

In Chapter 1, it was clarified that a PVDF film as an AE transducer was able to detect AEs generated by termite attack, though its sensitivity was lower than an AE transducer with a PZT element. However, higher sensitivity was necessary for the practical detection of AEs in wood construction, where there are inner posts or studs in the walls or joints. Lewis *et al*. (1991) and Lemaster *et al*. (1997) discussed the optimal resonant frequency for an AE transducer for detecting AEs generated by termite attack. They also pointed out the necessity of a waveguide, such as a lag screw attached to the AE transducer that penetrated walls to reach the inner posts or studs, for efficient detection of AEs generated in the walls.

It is necessary to effectively detect AEs generated by termite attack at the wider or deeper area in the practical wooden structures such as walls or joints.

In this chapter, a metal plate attached to the wood surface and a metal needle penetrated to inner posts or studs were employed as a waveguide to efficiently collect AEs and the feasibility of a plate- and needle-type waveguides for detection of termite attack was investigated.

2.2 Materials and methods

2.2.1 Detection of artificial AEs using plate-type waveguide

Western hemlock (*Tsuga heterophylla*), moisture content 10.2% and specific gravity 0.51, was cut into a specimen of 1000mm long with a cross section of 105×105 mm, and air-dried. Steel plates (15, 30, 45, and 60 mm square; 1, 2, 4, and 8 mm thick) were attached to the surface of the specimen with silicone-grease, as shown in Fig. 2.1.

AEs were detected by four cylindrical piezoelectric transducers (22 mm high, 8 mm in diameter; AE-901U, NF Corporation) with a resonant frequency of 140 kHz. One of the transducers was attached to the plate with silicone grease at a pressure load of 20 N to ensure close contact between the wood, the plate, and the transducer. The other three transducers were attached to the specimen around the steel plate with silicone grease and fixed with a rubber band. As the artificial AE source, a pencil lead (3 mm diameter, B-hardness) was broken 200 mm away from the center of the steel plate. The elastic waves generated in the specimen and transmitted to the transducer were transformed into a voltage oscillation using an AE measuring system (9640, NF Electric Instruments Co., Ltd.). Signals from the transducers were amplified by 40 dB and discriminated at a threshold voltage of 0.1 V to be recognized as an AE event. The characteristic AE data (peak-to-peak amplitude, duration time, rise time of each AE event, etc.) were collected and recorded on magnetic tape, and post-processed with a computer. Signals from the transducers and cumulative AE events were monitored by a digital oscilloscope (DL1200, Yokogawa Electric Corporation) and a pen recorder (CHINO Corporation), respectively.

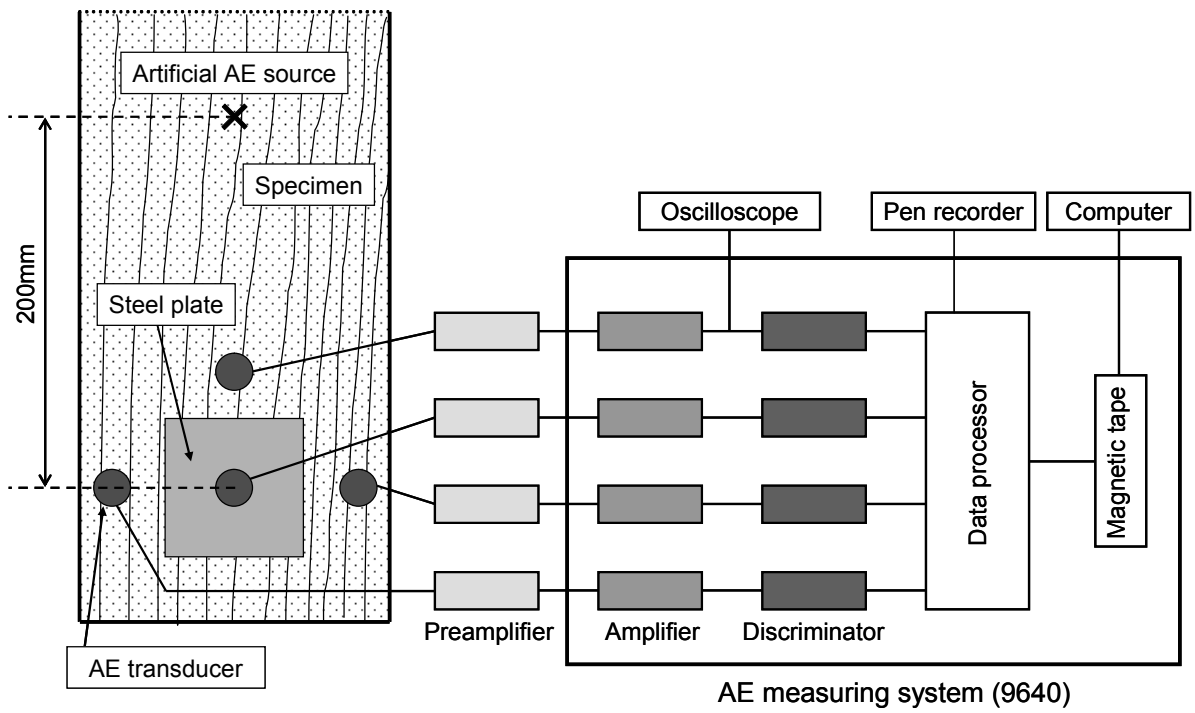

Fig. 2.1. Arrangement of AE transducers and AE measuring system

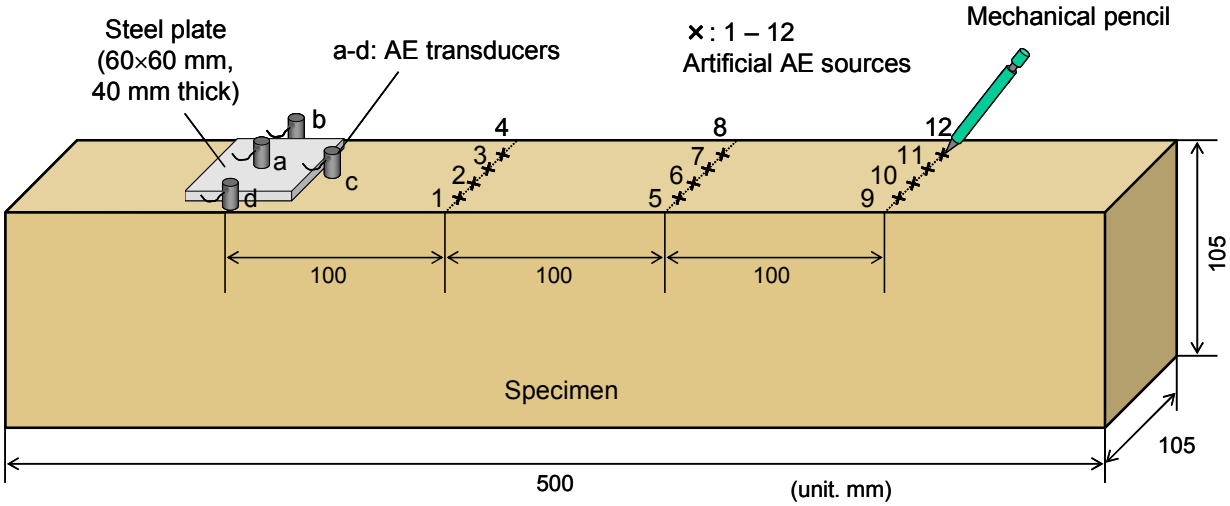

Fig. 2.2. Experimental setup of steel plate, AE transducers, and the locations of artificial AE sources.

A steel plate (60×60 mm, 4 mm thick) was attached to the surface of the specimen with silicone grease. As the artificial AE sources, a pencil lead was broken at four locations of each 100, 200, and 300 mm away from the center of the steel plate, as shown in Fig. 2.2. Signals from the transducers were processed and recorded in a procedure same as Fig. 2.1.

2.2.2 Detection of AEs generated by termite attack using plate-type waveguide

Western hemlock was cut into a specimen 500 mm long with a cross section of 105×105 mm, and air dried. A hole (10 mm in diameter and 30 mm deep) was drilled at the center of the end surface of the specimen. A group of 100 worker and 10 soldier termites of *Coptotermes formosanus* were collected from a colony maintained by the Research Institute for Sustainable Humanosphere, Kyoto University, had been kept starved for about two days was put into the hole, and the opening was then sealed with a silicone plug, as shown in Fig. 2.3.

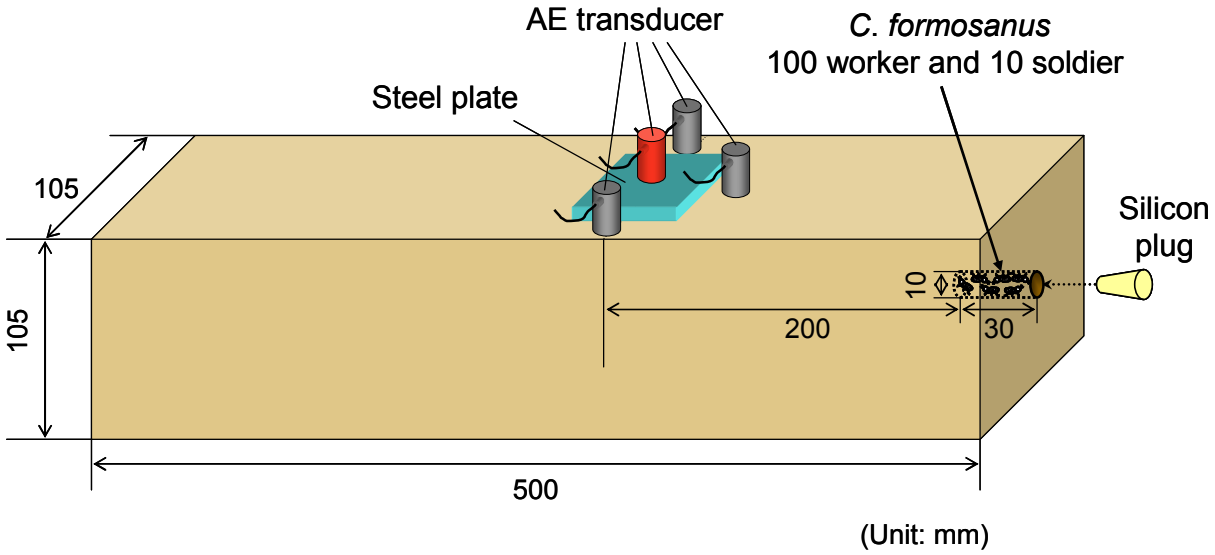

Fig. 2.3. Experimental setup for measurement of AE generated from termite feeding.

A steel plate (60×60 mm, 4 mm thick) was attached to the surface of the specimen with silicone grease. The distance from the center of the steel plate to the bottom of the hole was 200 mm. AEs generated by the feeding of termite were detected by the four transducers and measured under the same conditions as in Section 2.2.1, except that the signals from the transducers were amplified by 70 dB and discriminated at a threshold voltage of 0.26 V.

2.2.3 Detection of AEs generated by termite attack in wooden construction using needle-type waveguide

AE monitoring of termite attack on wood was applied to a house in Wakayama Prefecture in which wooden panel construction of suspicious for infestation of *C. formosanus*. AE monitoring was applied to the wall panel around a bathroom and the entrance porch and to the "Agarikamachi", a piece of wood at front edge of the

entranceway floor. The wall around the bathroom was made of wooden panels which were constructed of studs of two-by-four dimension lumber, covered with plywood and plasterboard, and finished with wall cloth. The Agarikamachi is made of glued laminated timber covered with a decorative veneer. It was easily conceirable that it would be difficult to detect AEs generated at the studs and the girths in the wall panel by sensors attached on the wall surface, because AE waves should attenuate strongely in the plaster board and the plywood. So an AE transducer with a resonant frequency of 140 kHz was attached to the wall surface using a needle-type sensor holder, which played a role of a waveguide penetrating through the wall covering made of plaster board, plywood, and cloth, as shown in Fig. 2.4. Signals from AE transducer were amplified by 85 dB, filtered using a high-pass filter of 50 kHz, discriminated at a threshold level of 0.6 V and recognized as an AE event.

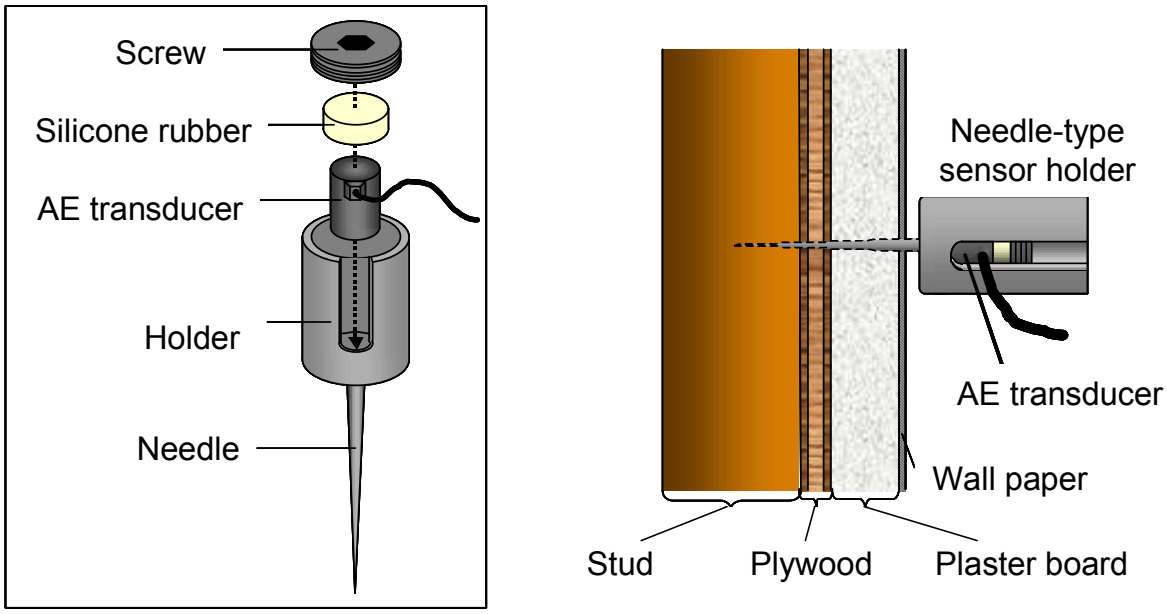

Fig. 2.4. Needle-type waveguide (left) and its setup into the wall panel (right).

2.3 Results and discussion

2.3.1 Detection of artificial AEs using plate-type waveguide

Figure 2.5 shows the average amplitudes of artificial AEs detected by an AE transducer attached to 16 steel plates as waveguides. It shows clearly that the larger the size of the plate is, the higher the average amplitude is, and that the thickness of the plate has no significant effect on the amplitude. The average amplitude of AEs detected by AE transducers attached directly to the wood was 4.42 V (Fig. 2.5). This suggests that an AE transducer with a plate larger than 30×30 mm can detect AEs more effectively than a transducer without a plate.

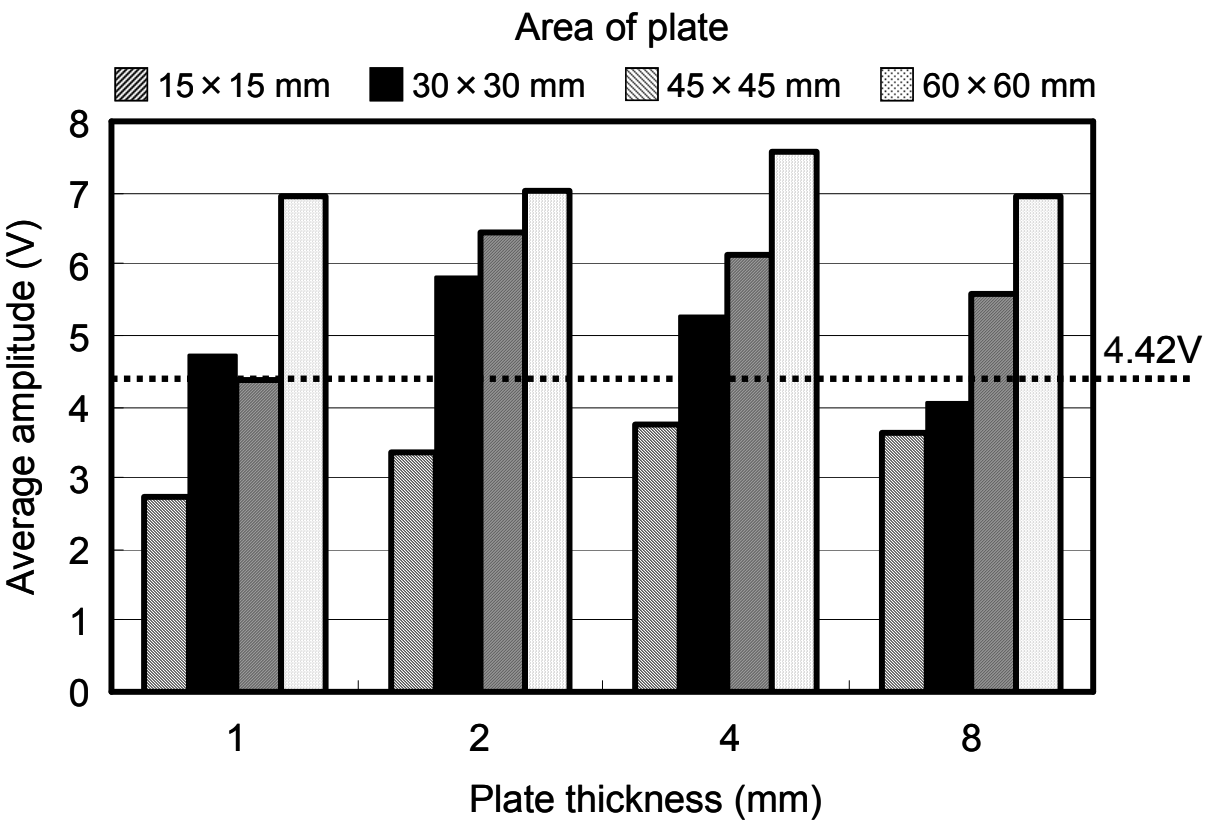

Fig. 2.5. Average of peak-to-peak amplitudes for detection with steel plates of different areas.

Dotted line shows average amplitude for detection without steel plate.

Artificial AEs generated at 12 locations ("1" to "12") on the surface of the specimen were detected by the four AE transducers ("a" to "d") with or without a steel plate (Fig. 2.2). Figure 2.6 shows the average of 10 peak-to-peak amplitudes of the detected AEs generated by breaking pencil leads for each transducer. For most cases, the average amplitudes decreased with increasing the distance between the AE transducer and the artificial AE source. Although the AE propagating distance between AE source "2" and transducer "a" is the same as that between "3" and "b", the amplitude for the former source-transducer combination was larger than that for the latter one. The amplitude can be influenced not only by the propagation distance but also by the propagation direction. This could be attributed to the facts that (a) AE waves were attenuated more when they propagated radially or tangentially than longitudinally, and (b) AE waves were attenuated with increasing the number of annual rings through which they propagated (Bucur *et al.* 1992). Although transducer "c" was located closer to the AE sources than transducer "a", which was fixed to the steel plate, the amplitudes for transducer "a" were the same as or larger than those for transducer "c". One possible explanation for this result is that the AE wave detected at transducer "c" was transmitted to the plate with only a little attenuation and detected by transducer "a". Another possibility is that the steel plate effectively detected AEs covering a wide area to result in large amplitudes from transducer "a". The above findings show the feasibility of using a steel plate as a waveguide to detect AEs propagating in wood, even if AE waves are more or less attenuated at the boundaries between the plate and the wood and between the plate and the AE transducer.

Figure 2.7 shows the relationships between the amplitude and the distance from the artificial AE source to the AE transducer for detection with and without a steel plate (60×60 mm, 4 mm thick). The amplitudes decreased with the distance and at distances of 100mm to 500mm the amplitudes for detection with a steel plate were about two to three times larger than without the plate. At distances more than 500 mm the steel plate was little beneficial as a waveguide and the amplitudes for detection with and without a steel plate were almost the same at a distance of 800 mm.

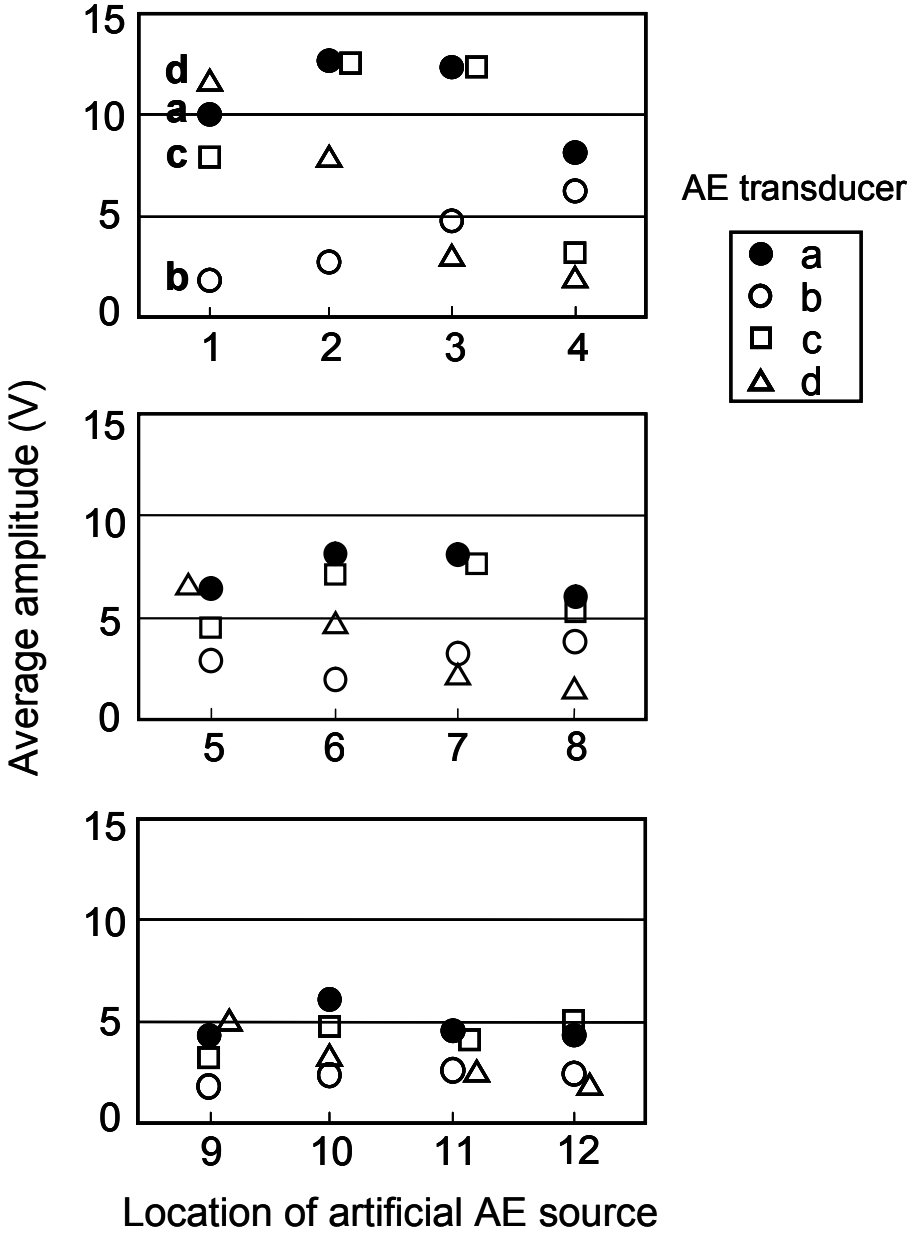

Fig. 2.6. Relationships between average of peak-to-peak amplitudes and location of artificial AE source.

AE transducers (a to d) and locations of artificial AE sources refer to Fig. 2.2.

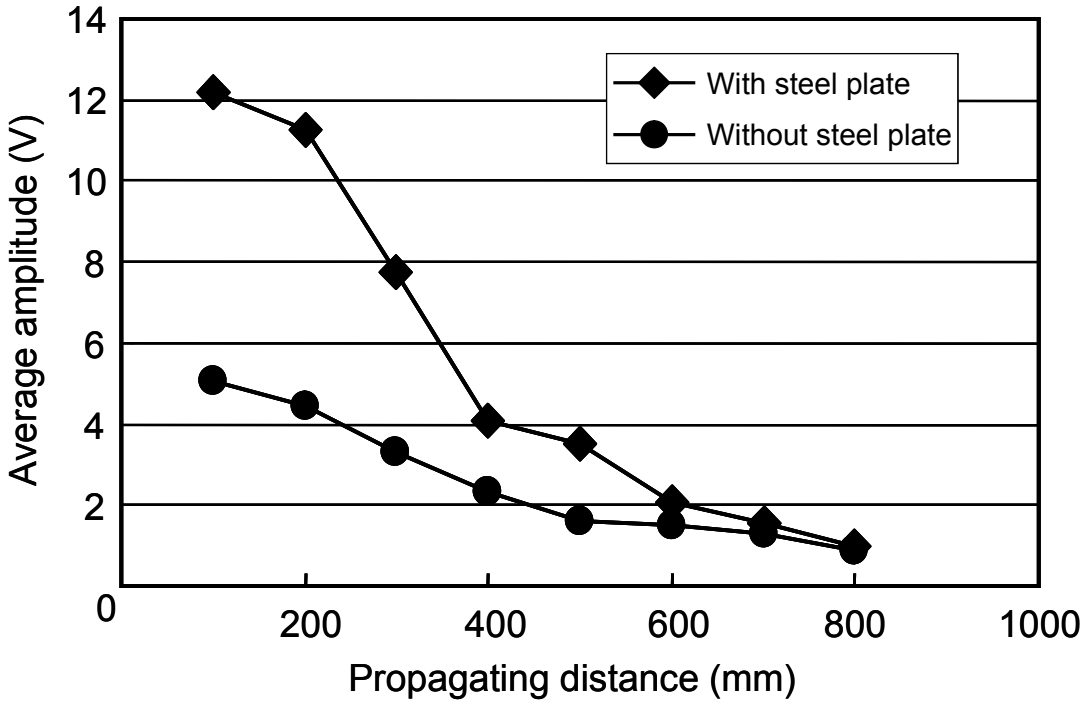

Fig. 2.7. Relationship between average amplitude and AE propagation distance.

2.3.2 Detection of AEs generated by termite attack using plate-type waveguide

Figure 2.8 shows cumulative AE event counts recorded during termite attack on wood for detection with and without a steel plate. "Burst-type" AEs were detected by the AE transducers with or without a steel plate, and the cumulative AE events increased with time. The AE transducer with a steel plate detected 2.5 to 10 times as many AE events as the AE transducers without a plate.

Figure 2.9 shows the amplitude distribution of the AEs detected over 180 minutes. The distribution had a peak near a threshold voltage of 0.26 V for both types of AE detection. The AE events detected with a steel plate were larger than those detected without a plate. A steel plate is expected to cover a wide area for the detection of AEs and help the AE transducer to detect weak and even attenuated AEs. This probably resulted in the higher count for detection with a plate. Therefore the AE event counts detected by AE transducer with steel plate were larger than without it.

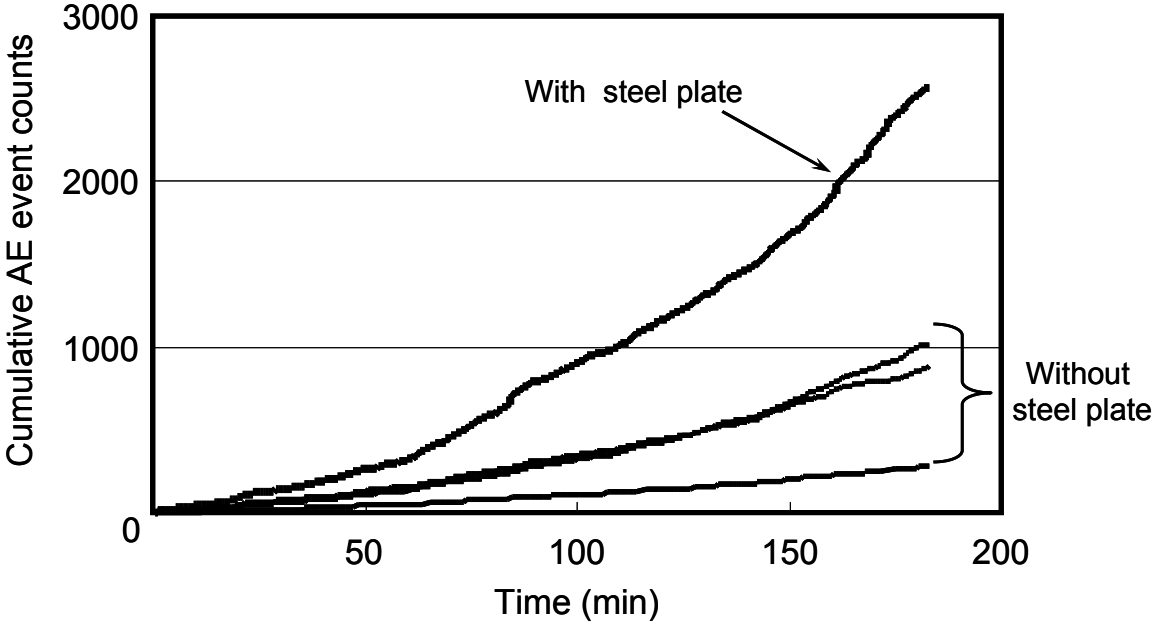

Fig. 2.8. Cumulative event counts of AEs detected with and without plate-type waveguide during termite attack.

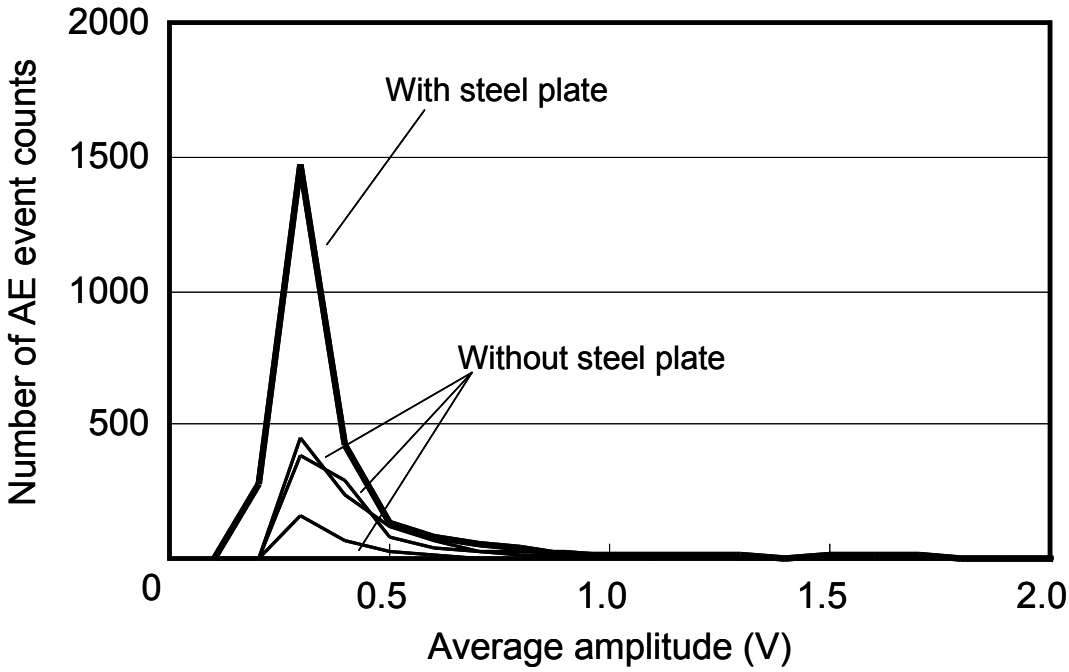

Fig. 2.9. Amplitude distribution of AEs detected with and without steel plate for termite attack.

2.3.3 Detection of AEs generated by termite attack in wooden construction using needle-type waveguide

Figure 2.10 shows AE event count rates per five minutes detected at the wall components of a wooden house using a needle-type waveguide. The waveguide penetrated to the stud columns and detected AEs at intervals of 300 mm. The AE event count rate was small in the upper part of stud columns S1 and S2 and no AE was detected in the stud columns S3, S4, and S5. The large rates (a maximum of 676) were detected in the girths sandwiched between wall panels of the first floor and the second floor, while no AE was detected at the stud columns in the wall panel of the second floor. These findings showed that the termite attack progressed from the stud columns toward the girths, not the second floor, and spreaded in the girths. Figure 2.10 also showed that AEs generated at the studs were effectively detected by using the needle-type waveguide in spite of the difficulty in propagating in the plaster board or plywood.

After the AE measurement, termiticide was injected at the point of the girth where the highest AE event rate was measured, and the cloth, plaster board, and plywood were carefully detached from the wall construction. It was found from the careful inspection that there were termite gallaries and dead bodies of termites in the area of higher AE event rate and the point that showed a maximum AE event rate of 676 was located near one end of the gallary network, where active termite attack should have been found (Fig. 2.11).

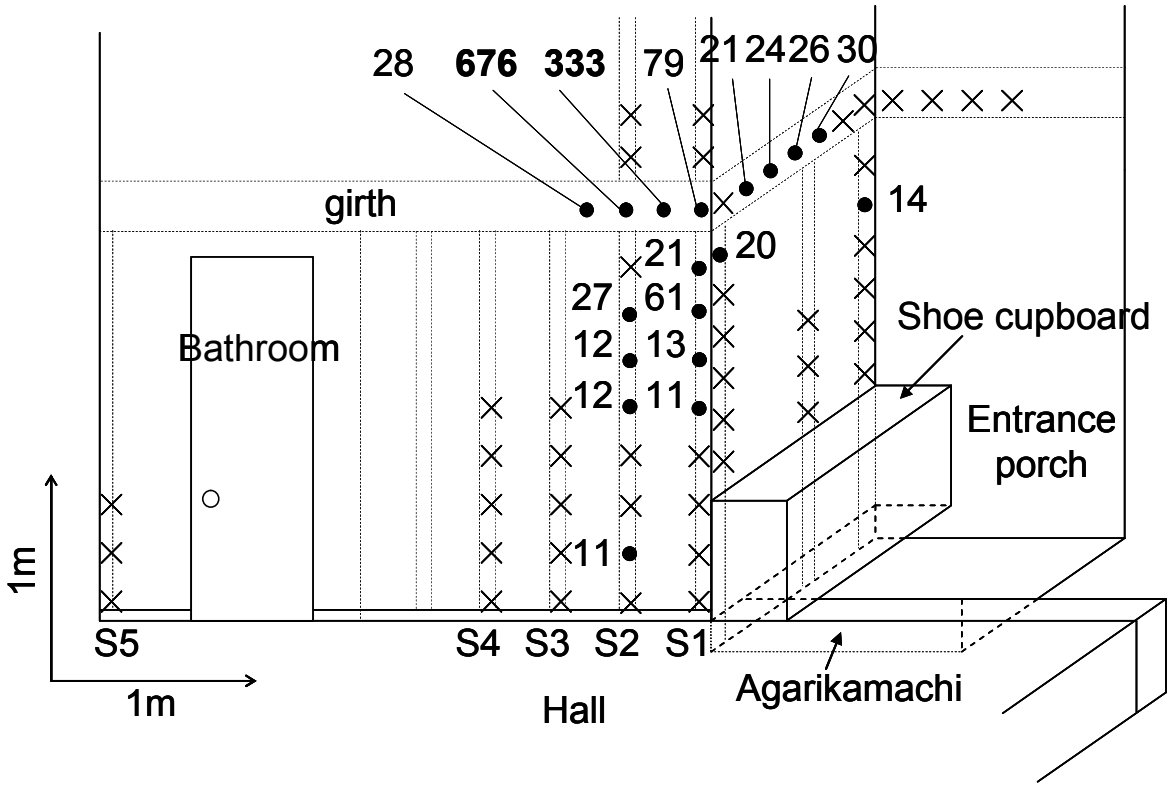

Fig. 2.10. AE event count rates per five minutes at the studs and the girth in the wall panel.

S1 to S5 are the stud columns in the wall. Blackened circles indicate the points where AEs were detected and the number shows the AE event count rates per five minutes. Cross marks indicate the points where no AE was detected.

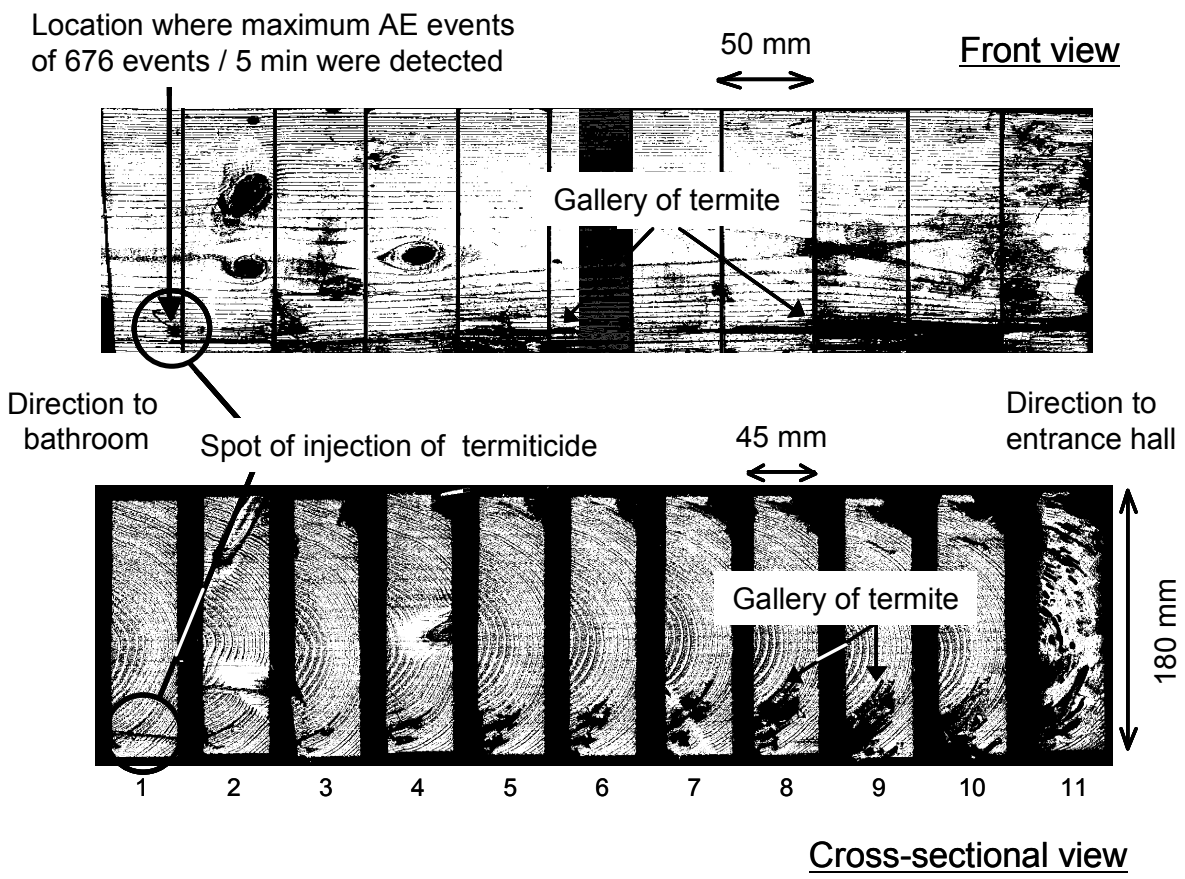

Fig. 2.11. Distribution of gallery of termite in the girth.

Figure 2.12 shows the AE event count rates detected on the agarikamachi at intervals of 150 mm using the needle-type waveguide. For the agarikamachi AEs were detected at AE event rates of 114, 41, and 34 at three points, and a rate of 3100 was measured at the joist in contact with the agarikamachi. This implies that termite attacks that had begun at the joists in the floor panels progressed toward the agarikamachi as well as studs and girths in the wall. The agarikamachi was disassembled after injection of the termiticide into the point of an AE event rates of 114. It was found from the careful inspection that there were termite gallaries and dead bodies of termites in the area of higher AE event count rates.

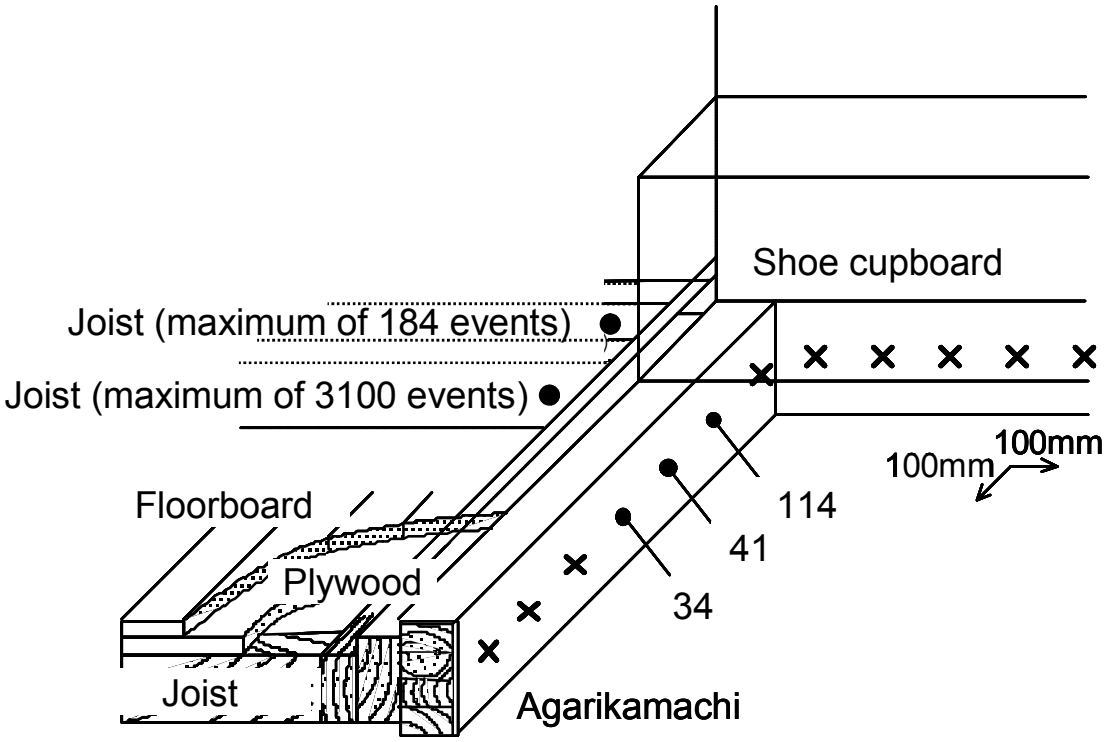

Fig. 2.12. AE event count rate per five minutes at the "Agarikamachi", piece of wood at front edge of entranceway floor.
Blackened circles with numbers and cross marks refer to Fig. 2.10

2.4 Summary

In order to examine the feasibility of using plate-type and needle-type waveguides for the effective detection of AEs from the termite attack in wood, AEs generated by breaking pencil leads and termite attack were detected using an AE transducer with a resonant frequency of 140 kHz with steel plates of four different sizes and thickness and three AE transducers without them. The larger area the plate had, it provided the larger amplitude for detection of the artificial AEs. The amplitudes of AEs detected by an AE transducer with a steel plate larger than 30×30 mm were greater than those without the plate for the artificial AE source. For detection of AEs generated by the

feeding of termites, *Coptotermes formosanus*, the cumulative AE event counts detected with a steel plate were much more than those without the plate. This shows that a steel plate is useful for detection of AEs generated in wood. Significant AE events more than 10 were successfully detected for five minutes from studs and from girth in the wall and glued laminated timber in the agarikamachi using a needle-type waveguide (sensor holder). It was confirmed after disassembling wall construction that the points where abundant AEs were detected were located at the places where termites were actively attacking wood. These findings suggest that it is quite useful for detection of termite attack in wood to employ a plate- and needle-type waveguides.

Part II

Detection of Termite Attack in Wood Using Semiconductor Gas Sensors

Chapter 3 **Detection of Metabolic Gas Emitted by Termites Using Semiconductor Gas Sensors**

Chapter 4 **Evaluation of Concentrations of Hydrogen and Methane Emitted by Termites Using Semiconductor Gas Analyzer**

Chapter 5 **Measurement of Hydrogen and Methane Emitted by Termites under Feeble Air Ventilation**

Chapter 3

Detection of Metabolic Gas Emitted by Termites Using Semiconductor Gas Sensors

3.1 Introduction

Trained dogs have been employed to detect odor components emitted from termite colonies (Lewis *et al*. 1997). It takes, however, much time and cost to train dogs, though trained dogs can detect odors from termites with an outstanding accuracy (Brooks *et al*. 2003). Therefore, it is necessary to develop an easy method, instead of method using trained dogs, to detect metabolic gas emitted from termites for the detection of early termite attacks in the wooden construction.

In this chapter, three types of semiconductor gas sensors (odor, methane, and hydrogen sensors) was employed to detect metabolic gases emitted by termites, and their feasibility for nondestructive detection of termite attacks in wooden structures was investigated.

3.2 Materials and methods

3.2.1. Experimental setup for gas measurement

Three types of semiconductor gas sensors provided by New Cosmos Electric Co., Ltd. with the selectivity to odor components, methane, and hydrogen were employed in this experiment. The sensor for odor components detects odor gas containing chemical compounds with molecular weight of fewer than 300, e.g. carboxylic acid, amine, or sulfuric compounds.

The basic operational principle of semiconductor gas sensor is as follows. A gas detection unit of the sensors made of tin oxide (SnO_2) is connected to a heater. In clean air, oxygen is adsorbed onto the surface of the unit in the state of negatively

charged O^-. When a combustible gas component accesses the unit heated at a fixed operating temperature between 300 and 450°C, it is combined with the adsorbed oxygen (oxidized) and the oxygen simultaneously releases an electron. This results in an increase in the electrical conductivity of the metal oxide, which is monitored as the decrease in the electrical resistance of the unit.

It was reported that using a small amount of another type of metal oxide as a catalyzer or changing the formation of SnO_2 and the heater, the gas detection unit got a function that selectively detected odor components, methane and other hydrocarbons (Suzuki and Takada 1995). The SnO_2 unit covered with a dense but fine porous silica (SiO_2) layer was employed to detect hydrogen (Katsuki and Fukui 1998), where only gases with the smallest molecular weight, such as hydrogen, can reach the gas detection unit due to the filtering function of silica.

A transparent container made of polypropylene with 110 mm inner diameter, 80 mm height, and 725 mL capacity was prepared, and three types of gas sensors were attached to the container lid (Fig. 3.1). A specific number of worker and soldier termites with and without food material (see next section) were placed in the container. The electrical resistance of the sensors during the detection of the gas components was monitored by a bridge circuit and a buffer amplifier, recorded to a voltage logger, and post-processed by a computer. The output voltage was converted into gas concentration by a calibration curve obtained in the preliminary experiment using the target gas (ethanol for odor sensor) with known concentration.

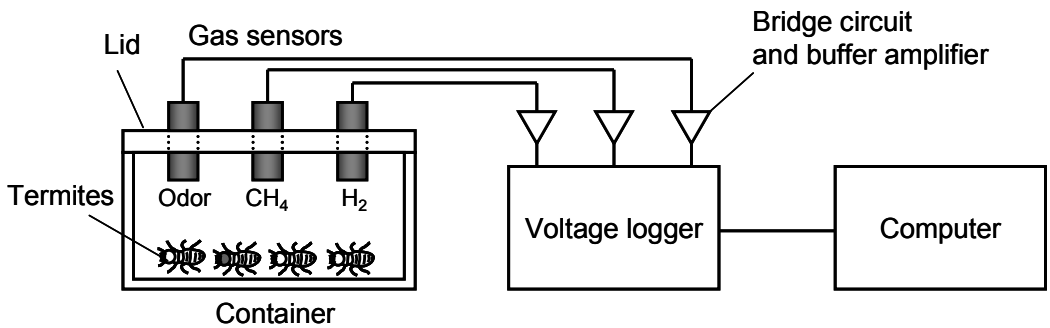

Fig. 3.1. Setup for gas measurement and data acquisition using three types of semiconductor gas sensors.

3.2.2 Conditions of gas measurement

Subterranean termites, *Coptotermes formosanus*, were collected from a colony maintained in the laboratory of the Research Institute for Sustainable Humanosphere, Kyoto University. Four termite groups with a constant ratio of the numbers of worker to soldier of 10 to 1 were employed: (1) 100 workers and 10 soldiers, (2) 200 workers and 20 soldiers, (3) 500 workers and 50 soldiers, and (4)1000 workers and 100 soldiers. Each termite group was put into a polypropylene container prepared for the following conditions (Fig. 3.2):

Condition 1: A sheet of wet filter paper was placed in the container.

Condition 2: A small wood specimen of Japanese red pine, *Pinus densiflora*, of 30(R) × 30(T) × 50(L) mm was moistened by water drops and placed in the container as food material.

Condition 3: A wet cotton sheet was placed under an acrylic tube with an 80 mm inner diameter and 50 mm height sealed by water permeable plaster at the bottom, and a small specimen of Japanese red pine was placed in the tube as food material.

Condition 4: Same as Condition 3, except that the termites had been kept starved for 2 days before gas measurement.

All gas measurements continued for 10 h after placing the termite group in the container. The gas measurement for each condition was repeated five times. The gas was measured without termites in each of the four conditions as a control.

The effect of the ratio of the numbers of worker to soldier on the gas concentration was also investigated using five termite groups: (1) 200 workers and no soldier, (2) 150 workers and 50 soldiers, (3) 100 workers and 100 soldiers, (4) 50 workers and 150 soldiers, and (5) no worker and 200 soldiers. These groups were placed in the polypropylene container under Condition 4 shown in Fig. 3.2 and after 10 h the gas concentration was measured using three types of gas sensors. The number of repetitions in the gas measurement was five.

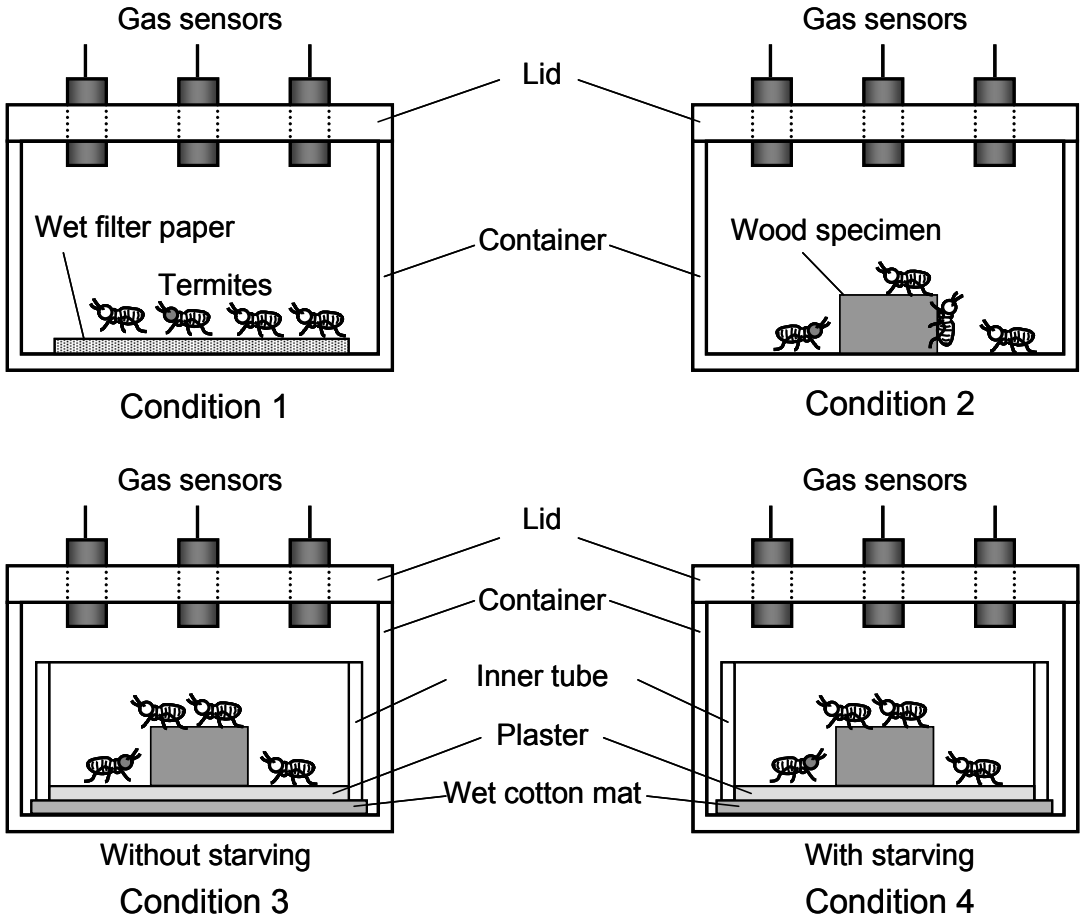

Fig. 3.2. Test conditions for detecting metabolic gas.

3.3 Results and discussion

Figure 3.3 shows an example of the time changes of the gas concentrations for a termite group of 500 workers and 50 soldiers in Condition 3. The gas concentrations increased for the first three hours and then little changed up to ten hours. For the methane sensor, the gas concentration was greater than 300 ppm from the beginning of the measurement, even though there was no methane source other than the termites in the container. This is probably due to an offset voltage in the gas detecting device, since the time change of methane concentration was similar among all conditions. Thus, the differences relative to the initial value are used in further discussion.

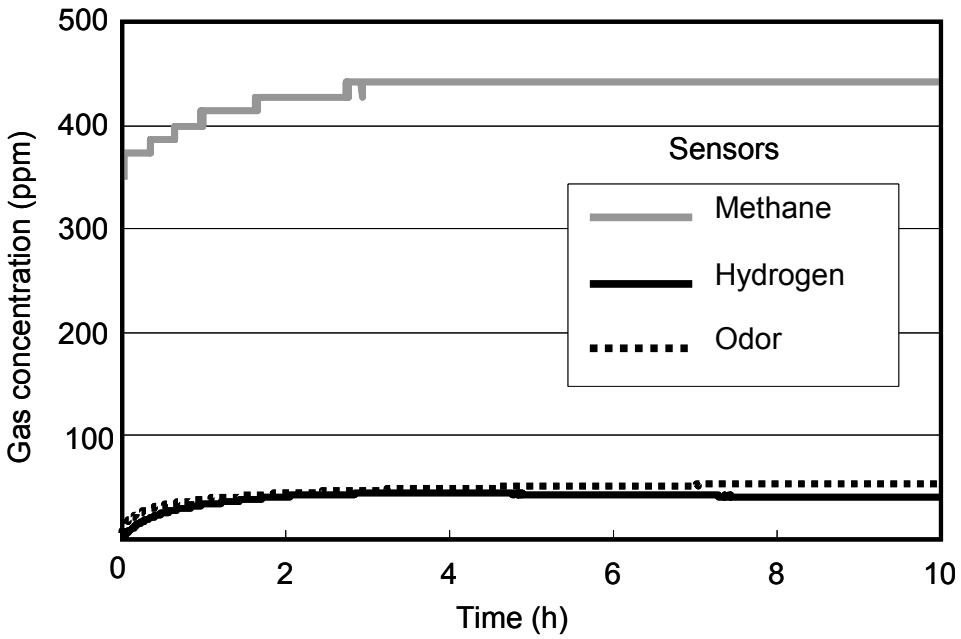

Fig. 3.3. An example of time change of gas concentrations measured by three gas sensors for a termite group of 500 workers and 50 soldiers in Condition 3.

The gas concentrations measured by odor, methane, and hydrogen sensors after 10 h are shown in Figs. 3.4, 3.5, and 3.6, respectively. For the odor sensor (Fig. 3.4), the concentrations were greater than 20 ppm for all conditions, except two controls (without food material) and Condition 1. There was a tendency that the concentration increased with the number of termites, but the correlation between them was not significant and the difference in the concentration between Conditions 3 and 4 (with and without starving) was not clear. In addition, more than 20 ppm of the odor component was detected for Japanese red pine without termites. These findings suggest that the gas concentrations measured by the odor selective sensor can be attributed mainly to the odor components emitted from the wood specimen. Thus, it was considered that the odor sensor was not suitable for the selective detection of odors emitted from termites.

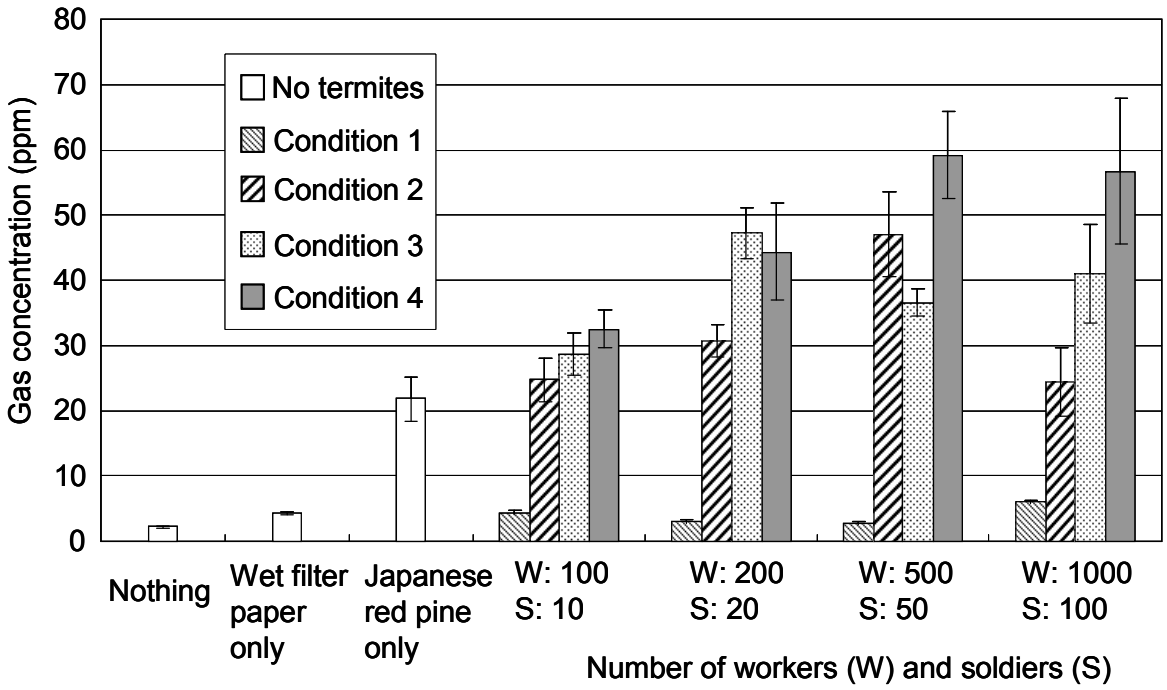

Fig. 3.4. Gas concentration detected by odor sensor after 10 h.
Error bars indicate standard deviation of five replications.

For the methane sensor (Fig. 3.5), the concentration under the conditions without termites was approximately 300 ppm, which can be attributed to the above-mentioned offset voltage in the device. The feeding of worker termites was expected to rise by addition of moisture (Condition 3) or by using the termites in a starved state (Condition 4). However, no significant increase in the concentration was observed in these conditions. Thus, it was difficult to detect methane generated from termite activity using the methane sensor, even though methane has been known as one of the most dominant metabolic gases emitted by termites (Khalil *et al*. 1990). Furthermore, the measurement period of 10 h may be too short to detect methane because the amount of wood consumption by termites was quite small during this period.

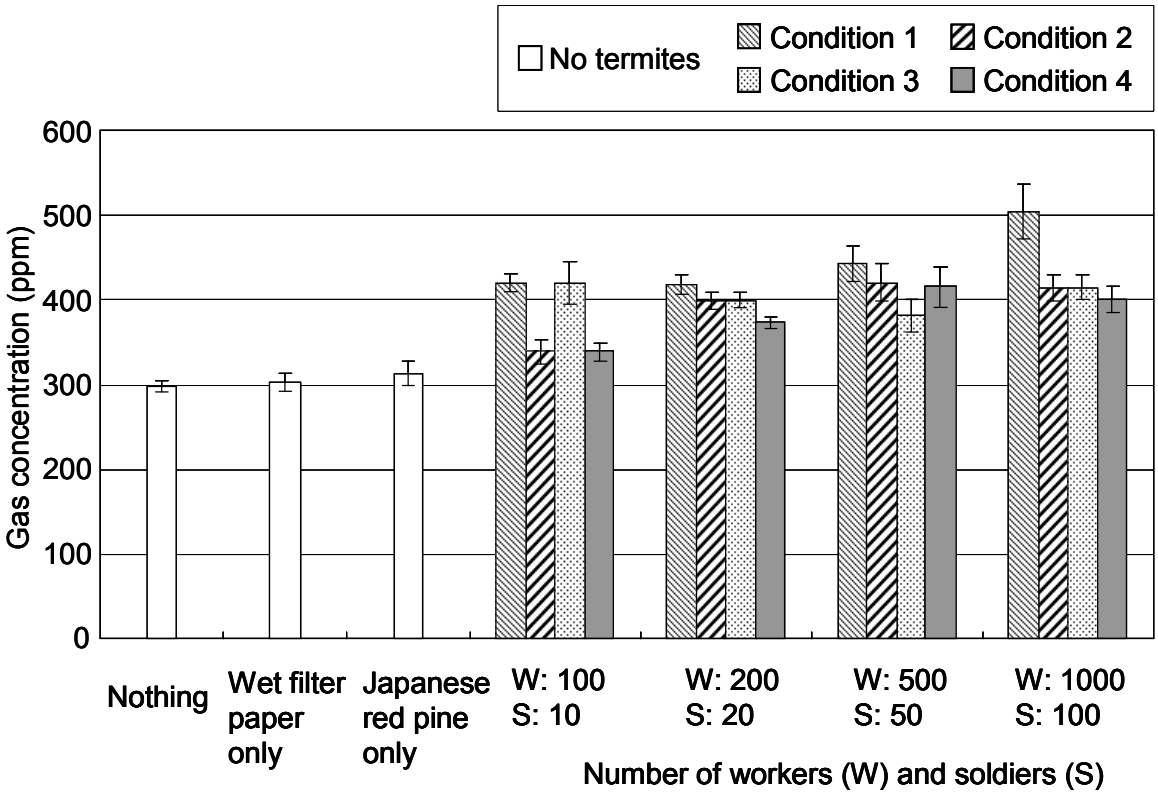

Fig. 3.5. Gas concentration detected by methane sensor after 10 h.
Error bars refer to Fig. 3.4.

For the hydrogen sensor, no gas was detected for the conditions without termites and the concentrations were measured to be 10–60 ppm for the conditions with termites (Fig. 3.6). The gas concentrations increased with the number of termites for all conditions except Condition 3. The gas concentrations for Condition 4 (with starving) were higher than those for Condition 3 (without starving) except for the case with 200 workers and 20 soldiers. The effect of gas emission from wet filter paper or wood specimen was found to be negligible. Therefore, the change in hydrogen concentration may be attributed to the termite activity. This is consistent with a previous report that hydrogen is produced in the guts of termites by a termite-symbiont system (Sugimoto *et al*. 1998). The hydrogen sensor may be useful in early detection of structural infestation of termites, since the sensor can detect hydrogen from even small number of termites.

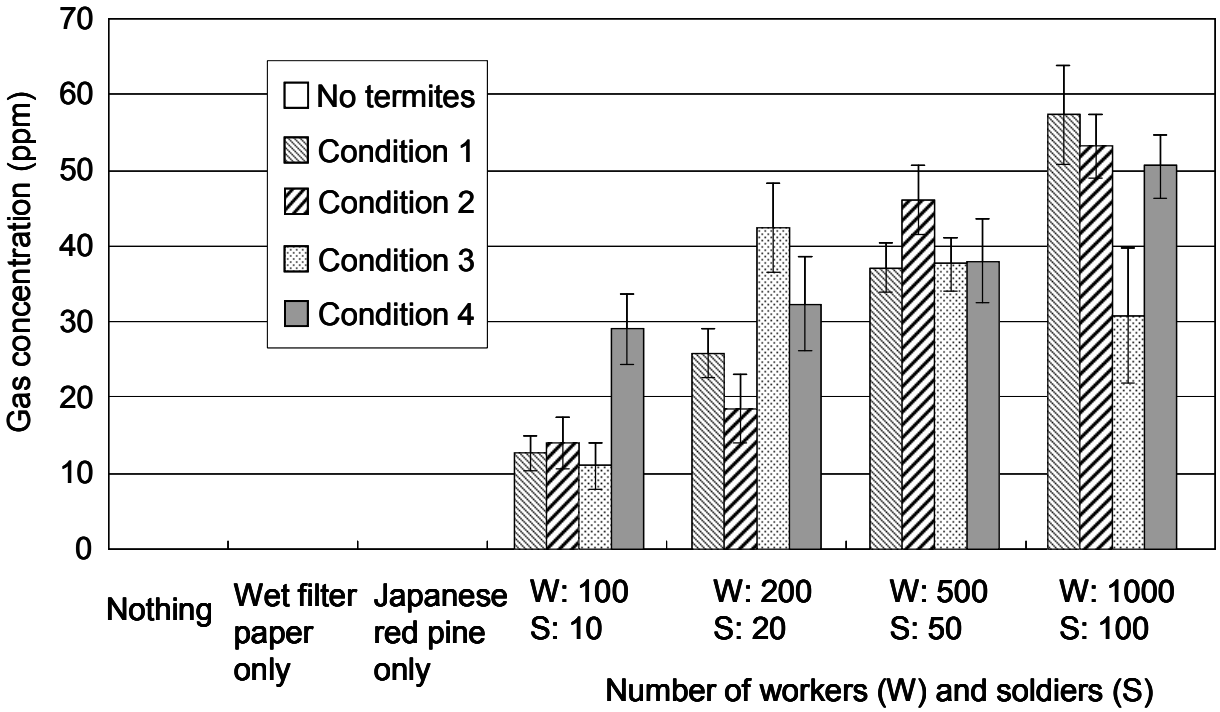

Fig. 3.6. Gas concentration detected by hydrogen sensor after 10 h.
Error bars refer to Figure 3.4.

Figures 3.7, 3.8, and 3.9 shows the concentrations measured by odor, methane, and hydrogen sensors, respectively, in Condition 4 for five termite groups with different worker-to-soldier ratios. No significant relationship was found between the ratio and the concentration of odor and methane, whereas the concentration of hydrogen was increased with the ratio of workers to soldiers. In particular, the hydrogen concentration was remarkably higher for groups of 200 and 150 workers than those for the group of 0 and 50 workers. This suggests that hydrogen was possibly generated by the feeding of workers, while soldiers that were provided with food material from workers emitted a small amount of hydrogen.

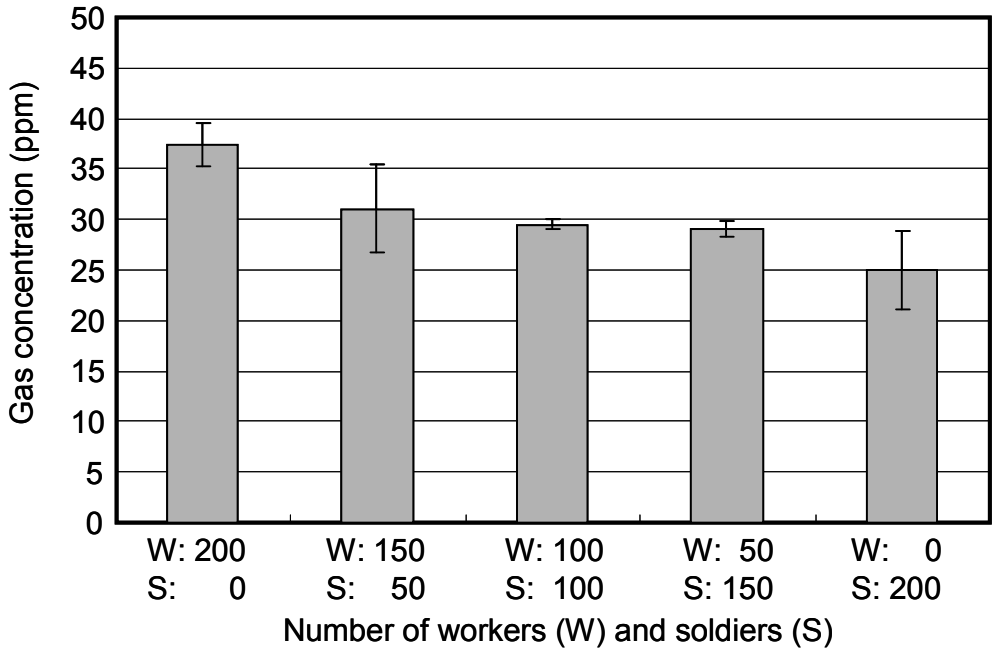

Fig. 3.7. Gas concentrations measured by odor sensor after 10 h in the condition 4 for five termite groups of different worker-to-soldier ratios.

Error bars refer to Fig. 3.4.

Fig. 3.8. Gas concentrations measured by methane sensor after 10 h in the condition 4 for five termite groups of different worker-to-soldier ratios.

Error bars refer to Fig. 3.4.

Fig. 3.9. Gas concentrations measured by hydrogen sensor after 10 h in the condition 4 for five termite groups of different worker-to-soldier ratios.
Error bars refer to Fig. 3.4.

3.4 Summary

The feasibility of three types of semiconductor gas sensors, odor-, methane-, and hydrogen-selective sensors made of tin oxide, for detection of the metabolic gas emitted by termites was examined. To investigate the relationship between the number of termites and the gas concentration and the effect of the existence of food material on gas production, five groups of different combination of workers and soldiers of *Coptotermes formosanus* were put into the polypropylene container with and without food material and the gas concentration was measured using three sensors after 10 h.

The hydrogen concentration increased with the number of termites and was not

influenced by the gases released from the wood specimen. Similar findings were obtained for the odor sensor, but it detected odor components from both termites and wood specimen. The methane sensor showed no significant increase in gas concentration for all conditions with termites. It was also found that the hydrogen emission depended mainly on the feeding of worker termites, while soldier termites made only a small contribution to hydrogen emission.

These findings suggest that the hydrogen sensor had the best performance for the detection of termite attacks on wood among three semiconductor sensors.

Chapter 4

Evaluation of Concentration of Hydrogen and Methane Emitted by Termites Using Semiconductor Gas Analyzer

4.1 Introduction

In Chapter 3, three types of semiconductor gas sensors were employed for detection of metabolic gases emitted by a small number of termites. The performance of the hydrogen sensor was found to be better than the odor and methane sensors. Hydrogen was emitted by the feedings of workers, while soldiers that were fed by workers emitted hydrogen a little. However, basic information on the performance of the gas sensor, especially for the quantitative estimation of the detected gas associated with the termite activity, remains unclear.

In this chapter, a specially designed gas analyzer equipped with a semiconductor gas sensor was applied for the nondestructive detection of termite attack in wood, and its feasibility was examined through qualitative and quantitative measurement of hydrogen and methane emitted by the termite feeding under several conditions.

4.2 Materials and methods

4.2.1 Termites and rearing conditions

A termite group composed of workers and soldiers were starved for two days and placed in a 450 mL glass bottle. The bottle had a metal lid with an opening (11 mm diameter) plugged with a butadiene rubber septum. The termites were held for 24 h in these tightly sealed bottles. A gas sample (2.5 mL) was aspirated by a syringe through the butadiene rubber septum, and the concentrations of hydrogen and methane were measured. Three experiments were conducted for the different termite groups:

Experiment 1: Subterranean termites, *Coptotermes formosanus*, were collected from a

colony maintained by the Research Institute for Sustainable Humanosphere, Kyoto University. Five termite groups of a constant ratio (10:1) of workers and soldiers in varying numbers were used; (1) 20:2, (2) 50:5, (3) 100:10, (4) 200:20, and (5) 500:50. Each termite group was put into a glass bottle with or without a small air-dried wood specimen of Scots pine (*Pinus sylvestris*) with a dimension of 35(R) × 35(T) × 5(L) mm. The glass bottles were sealed and placed in a thermohygrostat set at 28°C and 75% relative humidity (RH).

Experiment 2: 100 workers and 10 soldiers of *C. formosanus* were put into the glass bottle with or without a wood specimen of *P. sylvestris*. The bottles were placed in a thermohygrostat set at 5, 15, 25, 28, 35, and 45°C under a constant humidity of 75%RH for 24 h.

Experiment 3: Four termite species, *C. formosanus*, *Reticulitermes speratus* (subterranean termite), *Incisitermes minor* (drywood termite), and *Zootermopsis nevadensis* (dampwood termite), were used. *R. speratus*, *I. minor,* and *Z. nevadensis* were collected from a field colony at Kyoto University, a colony found in a wooden construction in Wakayama Prefecture, and a field colony from Hyogo Prefecture, respectively. Glass bottles with 20 workers of each species were placed in a thermohygrostat at 28°C and 75%RH for 24 h. A wood specimen of *P. sylvestris* of approximately 100% moisture content by soaking in water was put into the bottles for *C. formosanus*, *R. speratus*, and *Z. nevadensis*, and an air-dried flake of *P. sylvestris* was put in the bottle for *I. minor*.

Three bottles were prepared for each experiment for the replication of the measurement.

4.2.2 Measurement of gas concentration

A gas analyzer equipped with a semiconductor gas sensor was specially designed, developed, and used for qualitative and quantitative measurement of hydrogen and methane emitted by termite feeding (Fig. 4.1). A gas sample of 2.5 mL collected with a syringe was injected into an input unit. The sample was introduced into a column

filled with active carbon along with clean air. The gas components passed through the column with different retention times depending on the molecular size. Each gas component was detected by a semiconductor gas sensor made of tin oxide (SnO_2). The operational principle of gas sensors was the same described in Chapter 3.

As shown in Fig. 4.2, peaks recorded at retention times of 20 and 150 s corresponded to hydrogen and methane, respectively, and the sensitivity of the gas sensor for methane was lower than that for hydrogen. The recorded peak voltage was transformed into the gas concentration using the calibration curve obtained in preliminary experiments using the target gas of hydrogen or methane with a known concentration.

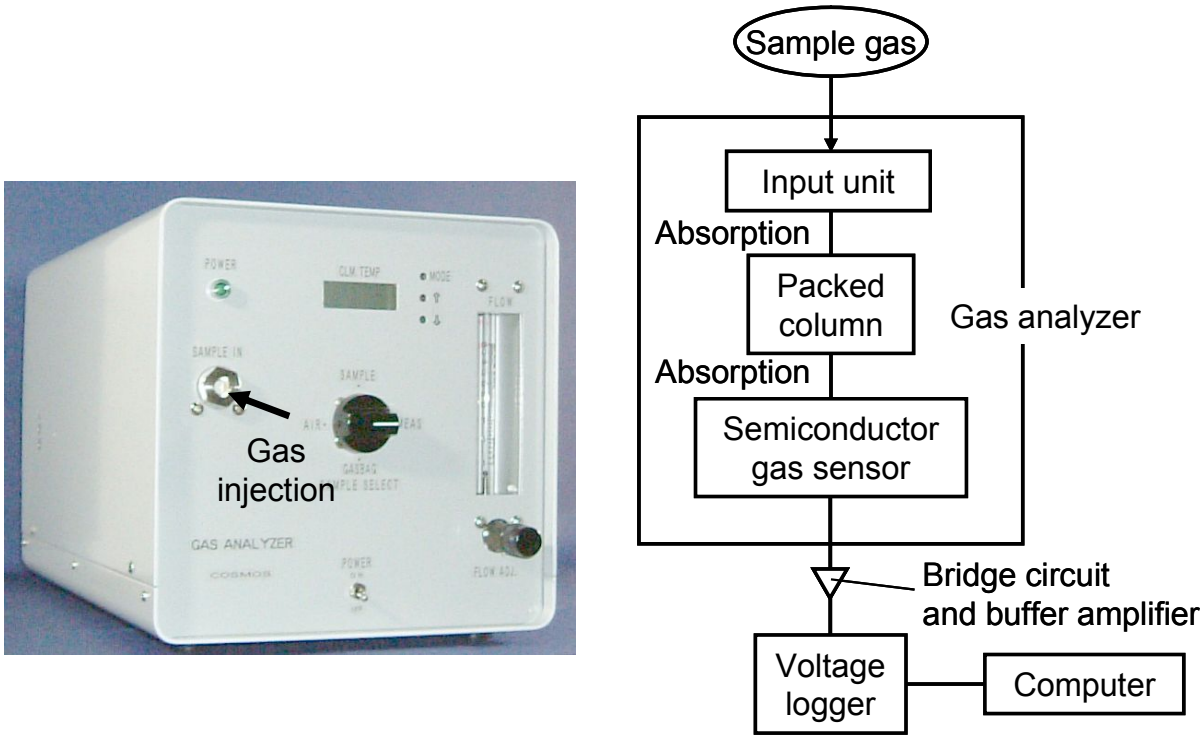

Fig. 4.1. Gas analyzer (left) and schematic outline of gas analysis (right).

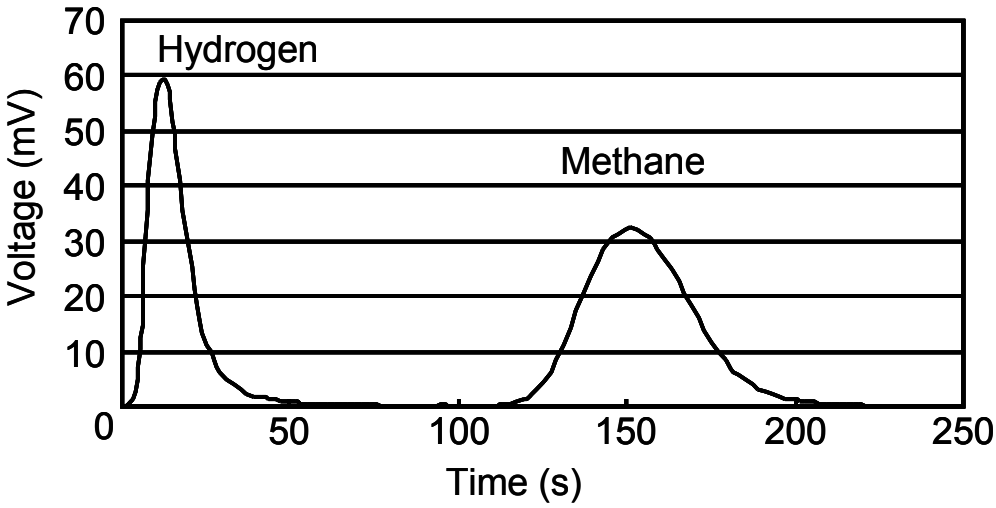

Fig. 4.2. Chromatogram for a mixture gas sample of 10 ppm hydrogen and 1000 ppm methane.

4.3 Results and discussion

4.3.1 Effect of the number of termites

The concentrations of hydrogen and methane measured in Experiment 1 are shown in Figs. 4.3 and 4.4. For all the termite groups, the concentrations were higher in the bottles with the wood specimen than those without it. This implies that hydrogen and methane were produced in the guts of worker termites by the termite-symbiont system and emitted in atmosphere (Sugimoto *et al.* 1998). That is, in other words, the gases were emitted by the feeding of worker termites. Significant levels of hydrogen and methane were also detected from the bottles without the wood specimen, although the concentration was very low. This was probably due to the emission of residual gas in the guts of termites after starving for two days. The concentrations of the both gasses increased with the number of termites in the bottle. These findings lead to a hypothesis that only worker termites emitted hydrogen and methane at a constant rate during feeding for 24 h, and this was supported by the finding in a preliminary

experiment that slight hydrogen and no methane were detected in the bottles with only soldiers. The gas emission per worker per unit time can be estimated from the concentrations for five groups with wood specimens in Figs. 4.3 and 4.4. The emission rates, as average of five termite groups, for hydrogen and methane were 0.405 ± 0.088 (S.D.) and 0.318 ± 0.056 nmol/h/worker, respectively.

It has been reported that the emission rate of hydrogen had large difference among the termite colonies and was affected by food materials, e.g., wood, wood flour, cellulose, and glucose (Kawaguchi *et al*. 2005, 2006; Inoue *et al*. 2007; Diba and Yoshimura 2010, Yoshimura 2011). It has also been reported that the maximum emission rate of methane emitted by *C. formosanus* was approximately 0.39 nmol/termite/h in Erlenmeyer flask test (Tsunoda *et al*. 1993a, 1993b). This is well consistent with the methane emission rate above.

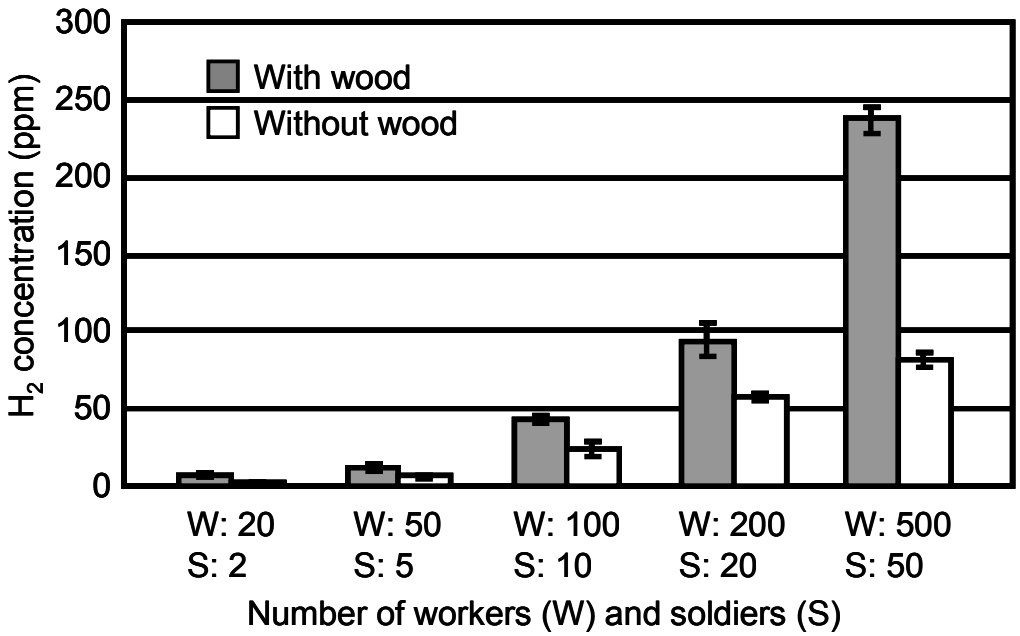

Fig. 4.3. Hydrogen concentrations for five termite groups of different number of workers and soldiers of *C. formosanus*.
Error bars indicate standard deviations of three replications.

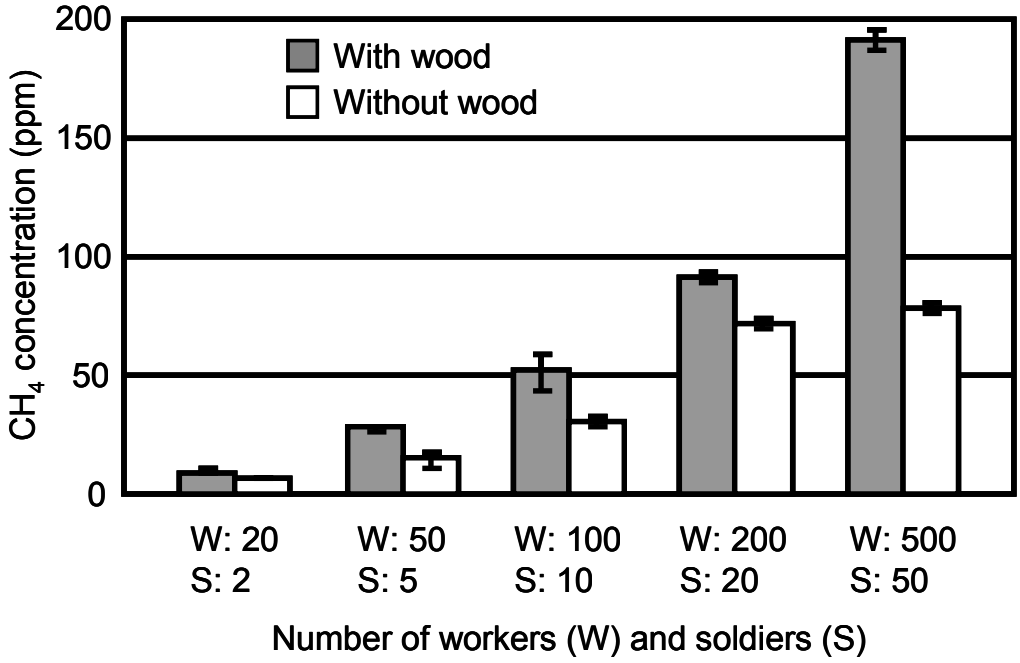

Fig. 4.4. Methane concentrations for five termite groups of different number of workers and soldiers of *C. formosanus*.
Error bars refer to Fig. 4.3.

4.3.2 Effect of ambient temperature

The concentrations of hydrogen and methane for different bottle temperatures in Experiment 2 are shown in Figs. 4.5 and 4.6. For both hydrogen and methane, the concentration was the highest at 35°C, which implies that the feeding of workers was the most active at this temperature. The average hydrogen- and methane-emission rates at 35°C were estimated to be 0.766 ± 0.131 (S.D.) and 0.441 ± 0.074 nmol/h/worker, respectively. At lower temperatures, 15 and 5°C, the concentrations of hydrogen and methane were less than 6 ppm and 15 ppm, respectively, and there was no significant difference in concentration with and without the wood specimen. This coincided with the observations that no active feeding was found at these temperatures. At 45°C the concentrations were low (less than 14 ppm) because all termites inside the bottle died within a few hours.

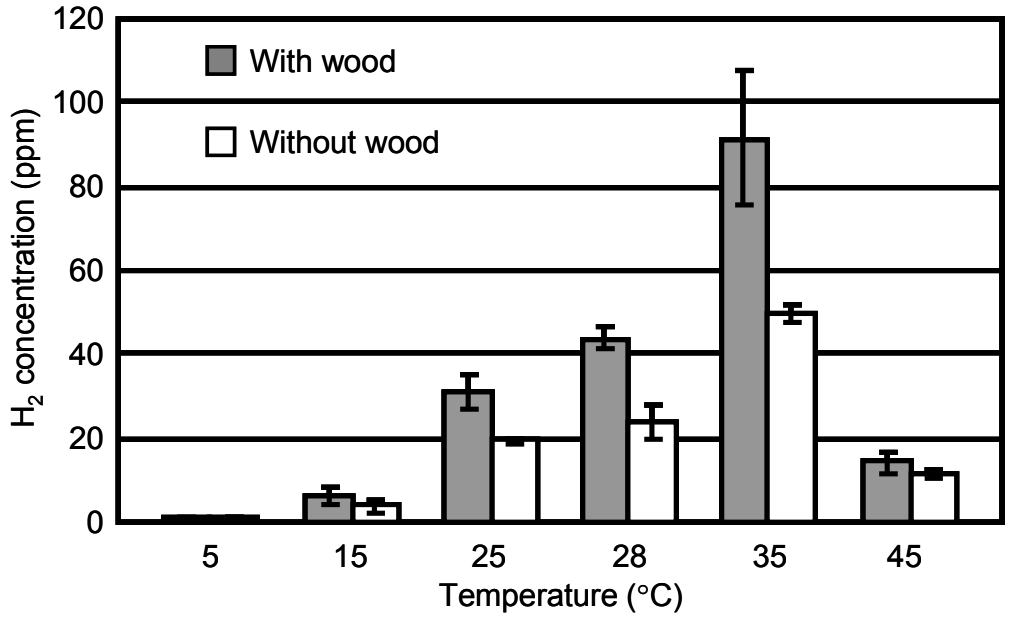

Fig. 4.5. Hydrogen concentrations for a termite group of 100 workers and 10 soldiers of *C. formosanus* under the six temperatures at 24 h after sealing.
Error bars refer to Fig. 4.3.

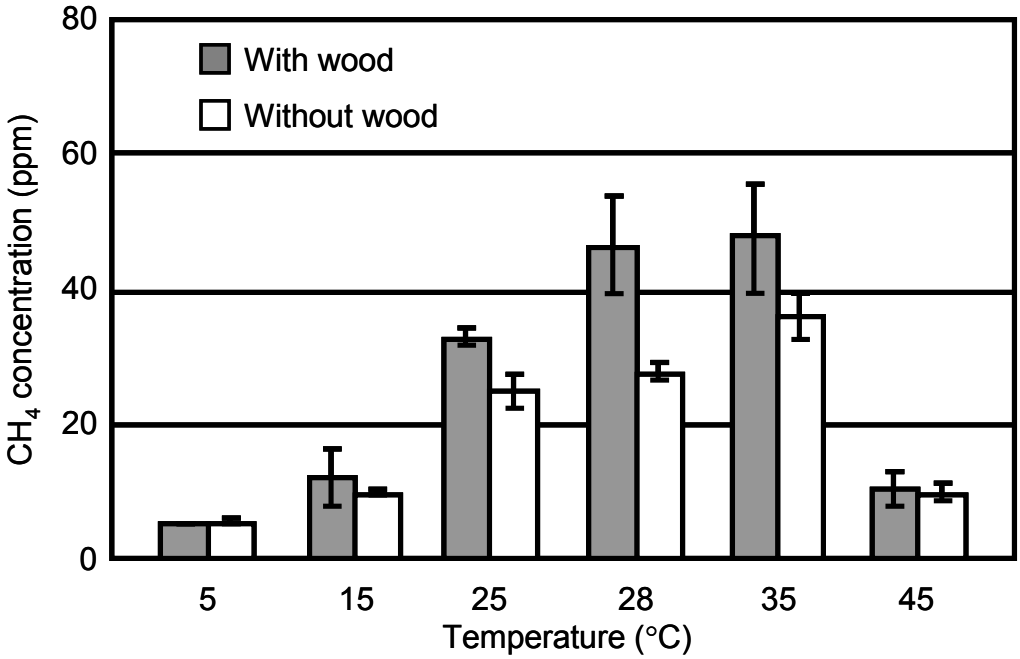

Fig. 4.6. Methane concentrations for a termite group of 100 workers and 10 soldiers of *C. formosanus* under the six temperatures at 24 h after sealing.
Error bars refer to Fig. 4.3.

It has been reported that the temperature in the nests of *C. formosanus* ranged from 5 to 35ºC throughout the year, and the largest number of AEs generated by the attack of worker termites in the wooden construction members were detected at 35°C (Yanase *et al*. 2003). This strongly supports the present findings.

4.3.3 Comparison among four termite species

The concentrations of hydrogen and methane for four termite species in Experiment 3 are shown in Fig. 4.7. A significant difference in the concentration of hydrogen and methane between the species was revealed by one way analysis of variance (ANOVA) [F (3, 8) = 7.59, $p < 0.01$]. The differences may be attributed to the difference in weight among four species, since the concentration of hydrogen was the highest for *Z. nevadensis* (average weight of 20 workers was 27 mg) and the lowest concentration was observed for *R. speratus* (average weight of 20 workers was 1.5 mg). On the other hand, the concentration of hydrogen was much lower (less than 4 ppm) for *I. minor* than *C. formosanus* (more than 10 ppm), although the average weight of 20 workers of the *I. minor* workers (5.2 mg) was greater than that of the *C. formosanus* workers (3.0 mg). This implies that not only the weight but also the feeding of termites possibly affects gas emission.

The concentration of methane was the highest for *Z. nevadensis* among four termite species. There was no distinct difference in concentration between *C. formosanus* and *R. speratus*. No methane was detected for *I. minor*. Sugimoto *et al*. (1998) reported that the probability of colonization by methanogens during the evolution of the termite-symbiont system may be lower in dry wood feeders than in wet and damp wood feeders, and the inhabitation of termite species in dry wood may also be responsible for the low probability of colonization by methanogens. Thus, the lack of methane detection for *I. minor* was probably because of the rearing and experimental conditions, feeding dry wood, and the fact that it is difficult to have colonization by methanogens without contact with soil.

As mentioned above, it is necessary to take the individual weight of worker

termites into consideration for estimation of gas emission rates. The gas emissions from a worker termite were estimated using the same method as in Figs. 4.3 and 4.4, and they were converted to the emission rates per individual weight of worker termites (Table 4.1). There was a significant difference in the emission rates of hydrogen and methane among four species [F (3, 8) = 7.59, $p < 0.01$]. The hydrogen- and the methane-emission rates were the highest for *C. formosanus* and for *R. speratus*, respectively. This implies that difference in the concentration of hydrogen and methane among termite species was affected by the metabolic system and the feedings of termite species (Zimmerman *et al.* 1982; Sugimoto *et al.* 1998). It is necessary to examine further these biological effects on the gas concentrations.

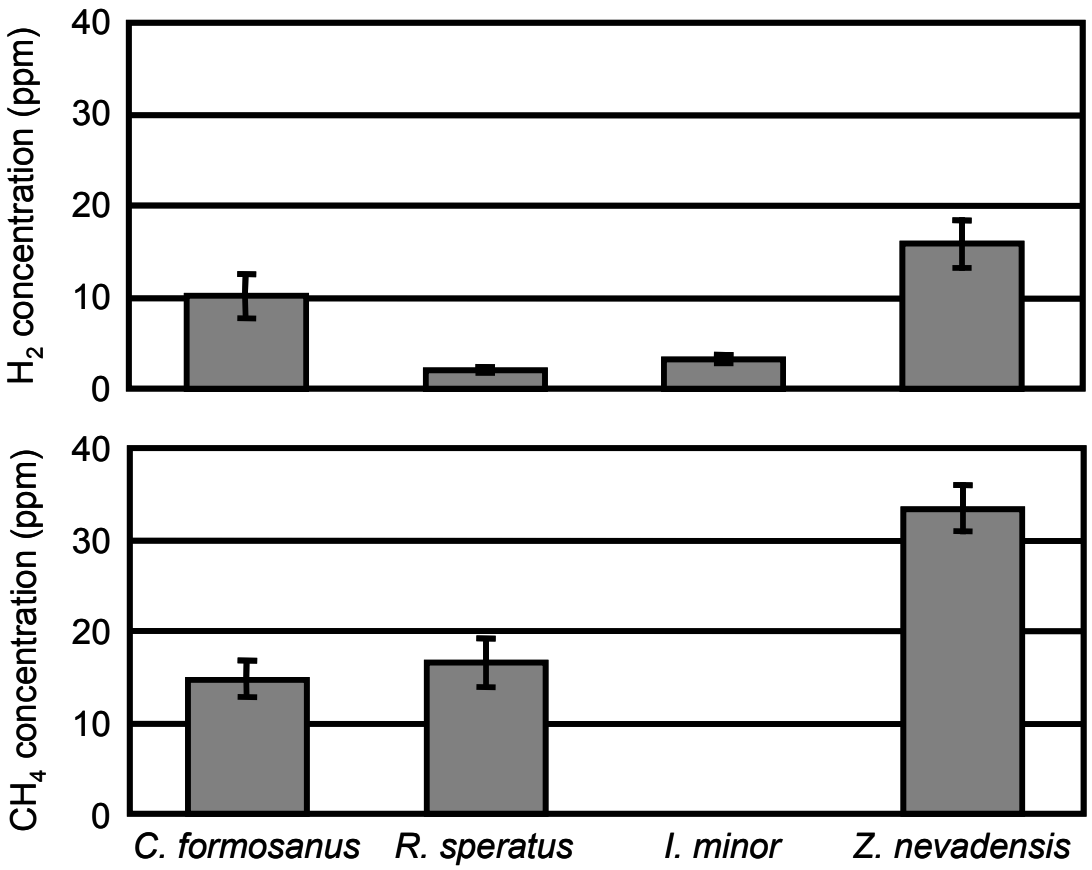

Fig. 4.7. Hydrogen and methane concentrations for 20 workers of four termite species. Error bars refer to Fig. 4.3.

Table 4.1. Emission rates of hydrogen and methane normalized to individual termite weight.

Termite species	Emission rate (nmol/h/mg)	
	Hydrogen	Methane
C. formosanus	0.139 ± 0.031	0.204 ± 0.029
R. speratus	0.056 ± 0.008	0.451 ± 0.077
I. minor	0.026 ± 0.002	ND
Z. nevadensis	0.024 ± 0.002	0.052 ± 0.002

ND, not detected

4.4 Summary

A gas analyzer equipped with a semiconductor gas sensor was employed for qualitative and quantitative measurement of hydrogen and methane emitted by termites. A gas sample of 2.5 mL was injected into the semiconductor gas sensor of the analyzer, and the maximum voltage of the sensor was converted into the gas concentration. The gas samples were collected from the bottles under three different conditions with termites; (1) five combinations of workers and soldiers of *Coptotermes formosanus* with and without a wood specimen; (2) *C. formosanus* under six temperature conditions; and (3) four termite species, *C. formosanus*, *Reticulitermes speratus*, *Incisitermes minor*, and *Zootermopsis nevadensis*. The hydrogen and methane concentrations increased with the number of termites. The gas concentrations were higher for the samples with a wood specimen than without it. Both hydrogen and methane concentrations were the highest at 35°C and were lowest at 15 and 5°C. The concentrations were very low at 45°C because all the termites had died in a few hours. The concentrations of hydrogen and methane were the highest for *Z. nevadensis,* a dampwood termite, among four species, and no methane was detected for *I. minor*, a drywood termite.

Chapter 5

Measurement of Hydrogen and Methane Emitted by Termites under Ventilation

5.1 Introduction

In Chapters 3 and 4, three types of semiconductor gas sensors (odor-, methane-, and hydrogen-selective) and a gas analyzer with a selective sensitivity to hydrogen and methane were applied for the detection of gases emitted by termites in a closed condition and some investigations were conducted from the viewpoints: the relationship between the number of termites and the gas concentration, the effect of the existence of food material on the gas production, the effect of the ambient temperature on the gas production, and the difference in gas concentration among four termite species. However, it was difficult to conduct a long term measurement of gas concentration in the closed condition, because the activity of termites decreased due to the lack of oxygen in about three days.

In this chapter, a gas measurement under forced ventilation at a feeble rate was conducted and the change in the gas emission during a long term measurement was examined to clarify the relationships between feeding of termite evaluated by AE generation and gas emission. Furthermore, the feasibility of gas detection method in the laboratory and practical situations was discussed.

5.2 Materials and methods

5.2.1 Long term measurement of hydrogen and methane under ventilation

Three termite groups of 200 workers and 20 soldiers of *Coptotermes formosanus* from the laboratory colony in the Research Institute for Sustainable Humanosphere, Kyoto University were put into a glass bottle of 450 mL with a metal lid. Three small holes with 11mm diameter were drilled at the lid for air flowing and gas sampling and

plugged by butadiene rubber septa, as shown in Fig. 5.1. For ventilation in the bottle, two silicone tubes were inserted into the bottle through the septa and ambient air was supplied into the bottle through the tube using air pump at 25 mL an hour. The bottle and the air pump were placed in a thermohygrostat set at 28 °C and 75 %RH. A sheet of wet paper as a food material was set in the bottles at 0 h, taken away at 48 h, and again set at 96 h. These procedures were repeated three times, as shown in Fig. 5.2. A gas sample of 2.0 mL was aspirated by a syringe through the septum and injected to a gas analyzer (Chapter 4), and the concentrations of hydrogen and methane were measured.

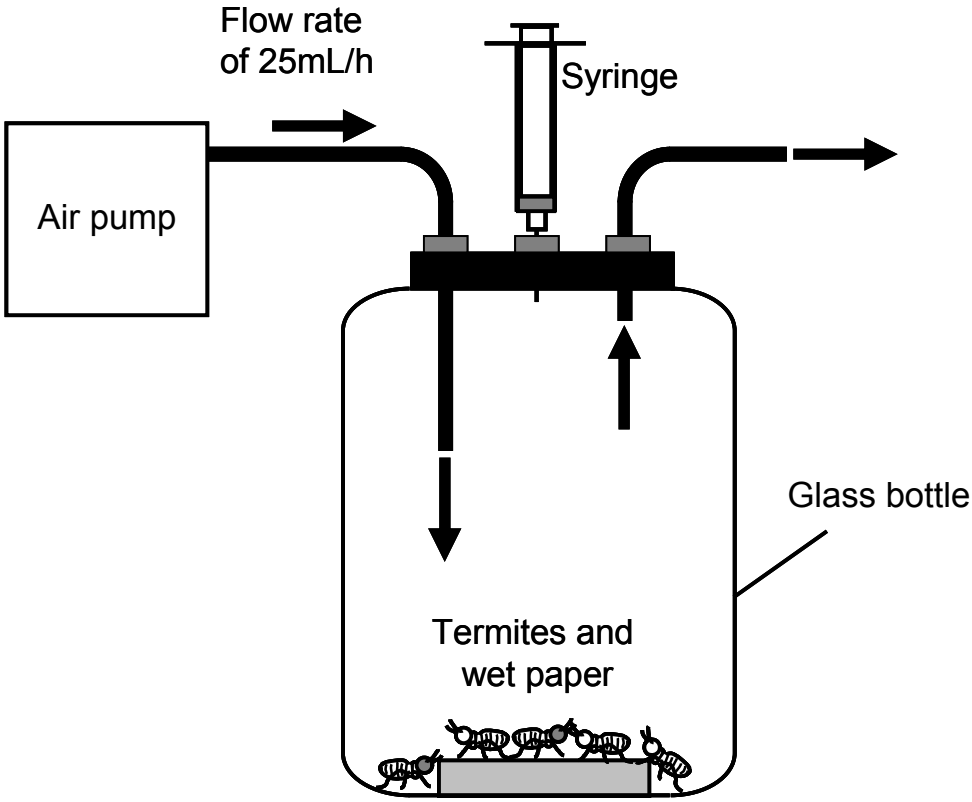

Fig. 5.1. Experimental setup for gas detection under forced ventilation.

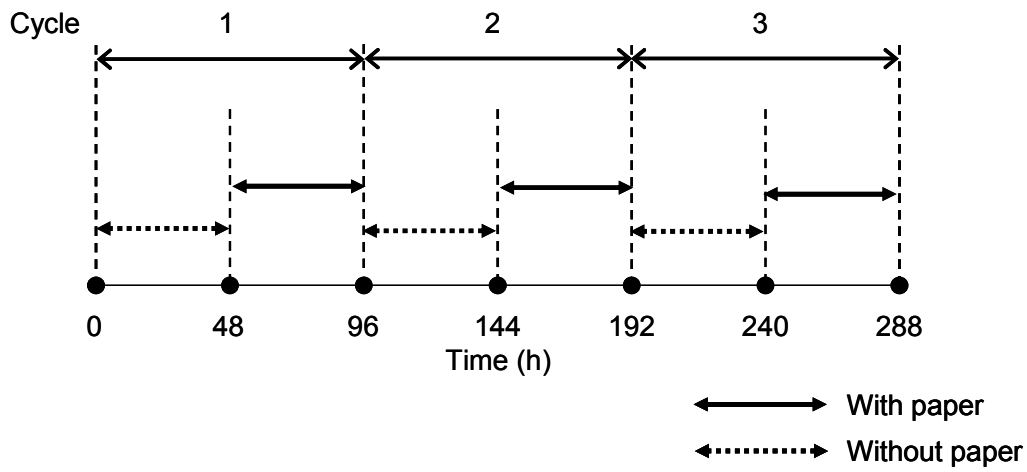

Fig. 5.2. The cycles of the condition in bottle with and without paper.

5.2.2 Simultaneous measurement of AE and gas concentrations

A termite group of 200 workers and 20 soldiers of *C. formosanus* from the laboratory colony were put into a glass bottle of 450mL with a metal lid, after termites were starved with only water for two days. Four small holes with 11mm diameter were drilled at the lid for air flowing, gas sampling, and AEs measurement and plugged by butadiene rubber septa, as shown in Fig. 5.3.

A gas sample of 2.0 mL was aspirated by a syringe through the septum and injected to a gas analyzer (Chapter 4). Ambient air was supplied into the bottle through the tube using air pump at 25 mL an hour. The bottle and the air pump were placed in a thermohygrostat set at 28 °C and 75 %RH. A food material with a size of 35 × 35 × 5(L) mm was prepared from scots pine (*Pinus sylvestris*), air-dried, and set in the bottle.

A cylindrical piezoelectric transducer (AE transducer, AE-901U, NF Corporation) with a fundamental resonant frequency of 140 kHz was attached to the surface of wood specimen with adhesive (Fig. 5.3). Signals from the transducer was filtered using a high-pass filter of 100 kHz, amplified by 66 dB and discriminated at a threshold voltage of 0.1 V to be recognized as an AE event, and the number of AE events was recorded continuously. Wood specimen with a transducer was placed on

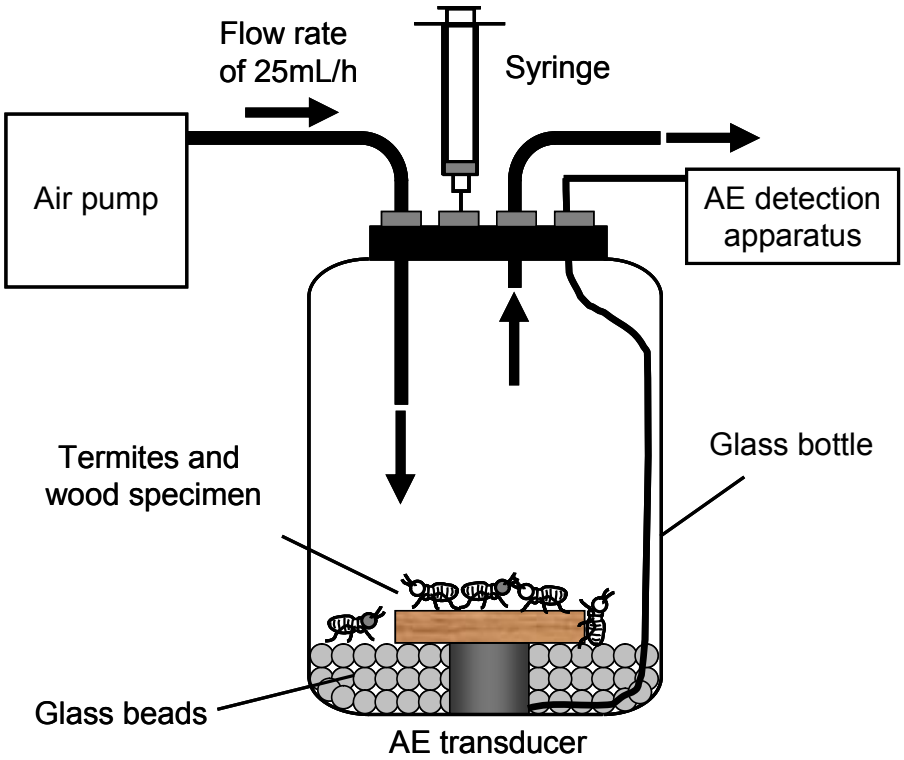

Fig. 5.3. Experimental setup for detection of gas and AE under forced ventilation.

the bottom of the bottle and a cable of AE transducer was pulled out of the bottle through the butadiene rubber septum. Glass beads were put in the bottle up to the top of the transducer so that termites can feed wood specimen easily.

5.2.3 Measurement of gas concentration under different ambient relative humidities

A termite group of 200 workers and 20 soldiers were put into an acrylic container of 500mL (100×100mm, 50mm height) with an acrylic lid. Four small holes with 11mm diameter were drilled at the lid for air flowing, gas sampling, and a temperature/humidity sensor and plugged by butadiene rubber septa (Fig. 5.4). Two silicone tubes were inserted in the container through two septa at the lid and the one was connected to an air pump for air flowing. A gas sample of 2.0 mL was collected from the container by aspirating using a syringe through the butadiene rubber septum

and the concentrations of hydrogen and methane were measured using a gas analyzer (Chapter 4). Three different termite groups were collected and employed; *C. formosanus* from the laboratory colony, *C. formosanus* from the field colony in the Living-Sphere Simulation Field, Kagoshima Prefecture, of Research Institute for Sustainable Humanosphere, Kyoto University, and *Reticulitermes speratus* from the field colony in Mt. Yoshida, Kyoto Prefecture. A group of 200 workers and 20 soldiers of *C. formosanus* and *R. speratus* without starving were put into the containers with a wood specimen of *P. sylvestris* of 35(R) × 35(T) × 5(L) mm, and the containers were placed in a thermohygrostat operated at 55, 65, 75, 85, and 95%RH under a constant temperature of 28°C. The air in the thermohygrostat was supplied into the container using an air pump at a flow rate of 25 mL/h. A gas sample of 2.0 mL was aspirated by a syringe through the septum every hour during 12 h after putting the termites into the container. The sample was injected to a gas analyzer (Chapter 4), and the concentrations of hydrogen and methane were measured. They were also measured at 24 h after the beginning of experiment. The gas measurements for each condition were repeated five times.

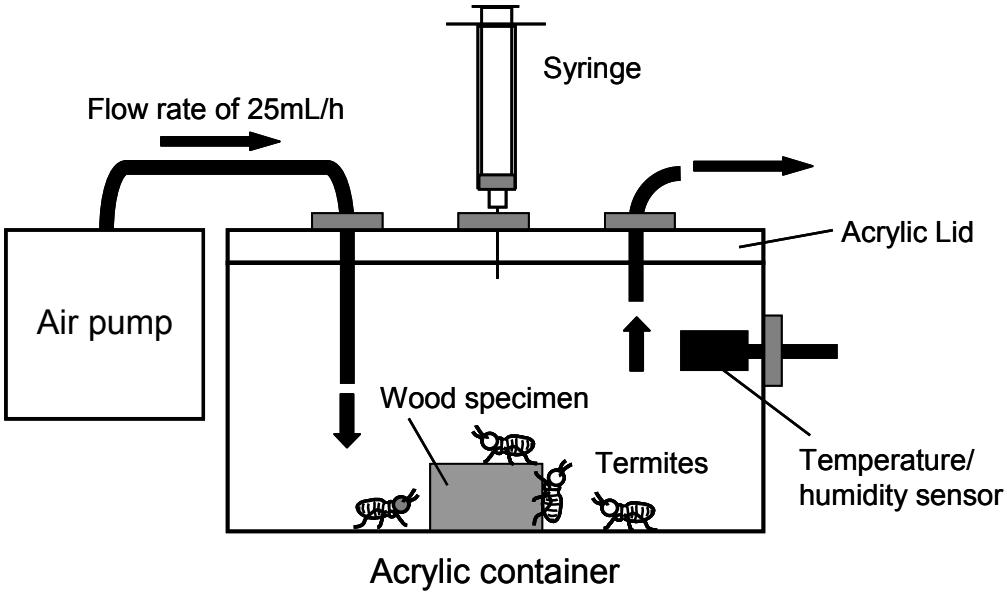

Fig. 5.4. Experimental setup for measurement of gas concentration under different relative humidities.

5.3 Results and discussion

5.3.1 Long-term measurement of hydrogen and methane

The time courses of the concentration of hydrogen under the air ventilation in bottle with and without paper were shown in Fig. 5.5. There is no figure for methane because the concentration of methane was too low to be detected using the gas analyzer. Figure 5.5 shows that the termite activity was maintained for 12 days. This is probably due to forced ventilation, because the termite activity decreased for about 3 days under the closed condition without ventilation. The concentrations were the highest at 4 h after inserting a paper in the bottle and then quickly decreased. The concentration was lower for without a paper than with paper, but it was relatively high immediately after taking the paper off, which may result from hydrogen emission by termites during digesting a paper in the guts.

Figure 5.6 shows the time changes of the hydrogen concentration that was the average of the corresponding plots at every 96 hours for Group 3 in Fig. 5.5. As shown in Fig. 5.6, the difference in concentration between bottles with and without paper for the first 12 h was much larger than that from 12 to 48 h. The hydrogen emissions from a worker per unit time can be estimated from the concentrations in Fig. 5.6 as follows:

$$\frac{(450-25)C_n + A_{n+1}}{450} = C_{n+1} \tag{1}$$

$$A_{n+1} = 450C_{n+1} - 425C_n \tag{2}$$

where C_n is the concentration of hydrogen at time T_n, C_{n+1} is the concentration of hydrogen one hour after time T_n, and A_{n+1} is the amount of hydrogen emitted for one hour from time T_n, considering the bottle capacity of 450 mL and ventilation of 25 mL an hour. The maximum emission rate of hydrogen for termite group C in Fig. 5.6 was 0.487 nmol/h/worker for with paper and 0.238 nmol/h/worker for without paper.

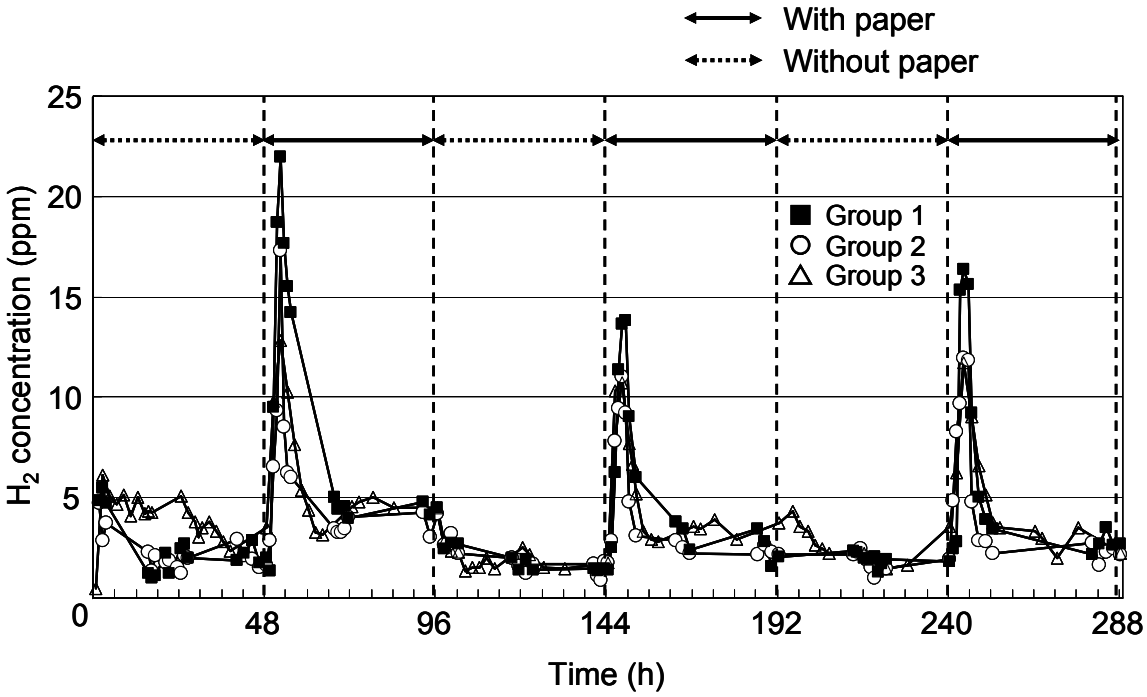

Fig. 5.5. Hydrogen concentrations during the three cycles of with and without paper for three termite groups.

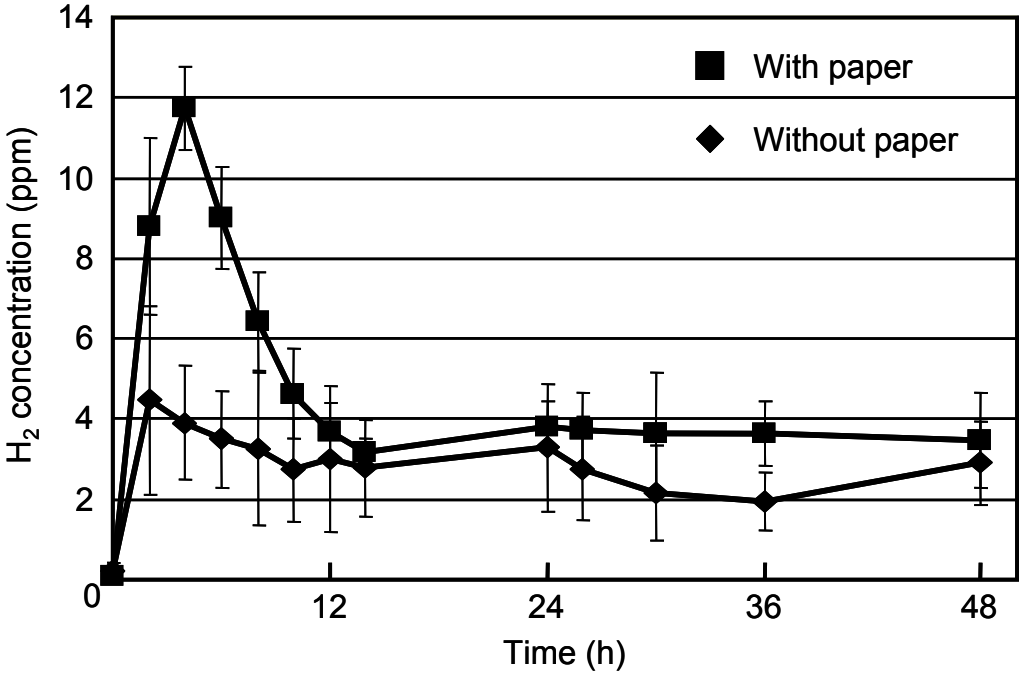

Fig. 5.6. Average concentration of hydrogen during three cycles for Group 3, refer to Fig. 5.5, with and without paper.

5.3.2 Relationship between AE and gas concentration

The time changes of AE event rate and the concentration of hydrogen emitted from termites were shown in Fig. 5.7. The sensitivity of the analyzer for methane was not so high that methane was not detected significantly under forced ventilation. There was a tendency that the concentration curve showed peaks a few hours later than those of AE event count rate. This implies that there was a delay between the feeding of termites, which was detected as AEs, and the hydrogen emission by termites due to the digestion of wood in their guts.

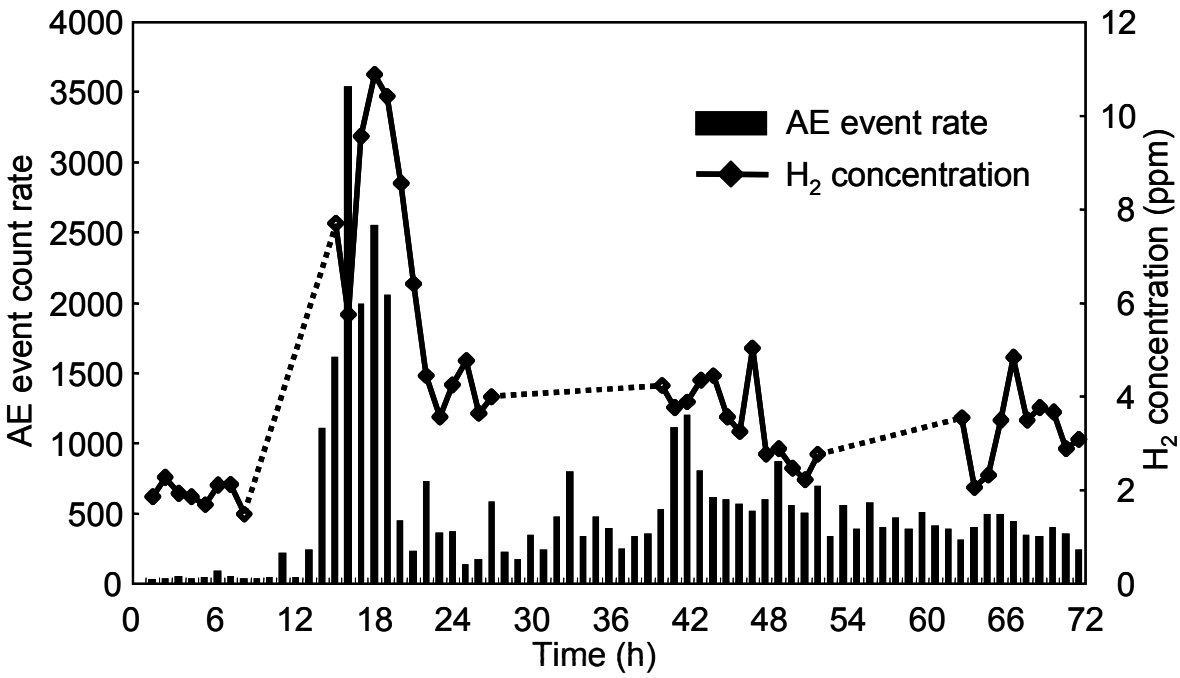

Fig. 5.7. Time changes of AE event count rate and hydrogen concentration under forced ventilation.

5.3.3 Effect of ambient relative humidity

Figure 5.8 shows that time changes of the hydrogen concentration at 75%RH for five termite groups. The concentrations were relatively stable for 24 h, though they fluctuated with a range of 2 ppm. Similar findings were obtained for other relative humidities. The hydrogen concentrations under five humidity conditions at 24 h from the beginning of each experiment were shown in Fig. 5.9, 5.10, and 5.11 for *C. formosanus* from the laboratory and the field colony and *R. speratus* from the field colony, respectively. The concentrations were the highest at 75%RH for three termite groups. This implies that the feedings of workers were the highest at this humidity. It has been reported that AE generations by the attacks of *C. formosanus* (Yusuf *et al*. 2000) and *R. speratus* (Nakayama *et al*. 2002, 2004) were the highest at 75%RH and at 70 and 80%RH, respectively. This supports the present findings. For all humidity conditions, the gas concentrations were higher for *C. formosanus* than for *R. speratus*, which is the same as obtained in Chapter 4. The concentrations were the lowest at 55%RH, especially for *R. speratus*, and this implies that it was difficult for subterranean termites such as *C. formosanus* and *R. speratus* to feed wood specimen at 55%RH or lower humidity. The concentrations for *C. formosanus* from the field colony were higher than those from the laboratory colony, in particular at 55 and 65%RH. The relative humidity in the room of the laboratory colony was kept at a constant of 28°C and between about 60 and 80%RH throughout the year. Therefore, the termites from the laboratory colony may be less adaptable to the climate change than those from the field colony inhabiting in the greater humidity change.

Methane was successfully detected for *R. speratus* as shown in Fig. 5.12, while it

was difficult to detect it for *C. formosanus*. The methane concentration for *R. speratus* was the highest at 75%RH and the lowest at 55%RH, which was the same as for hydrogen. The fact that for *R. speratus* it was possible to detect methane even under forced ventilation may support the finding that *R. speratus* showed the highest emission rate of methane (Table 4.1).

These findings suggest that there is a possibility to apply the gas measurement for the monitoring of termite infestation in the monitoring station where the ventilation is more or less inevitable condition (Su *et al*. 2000; Su 2001, 2002) and in the wall of wooden construction.

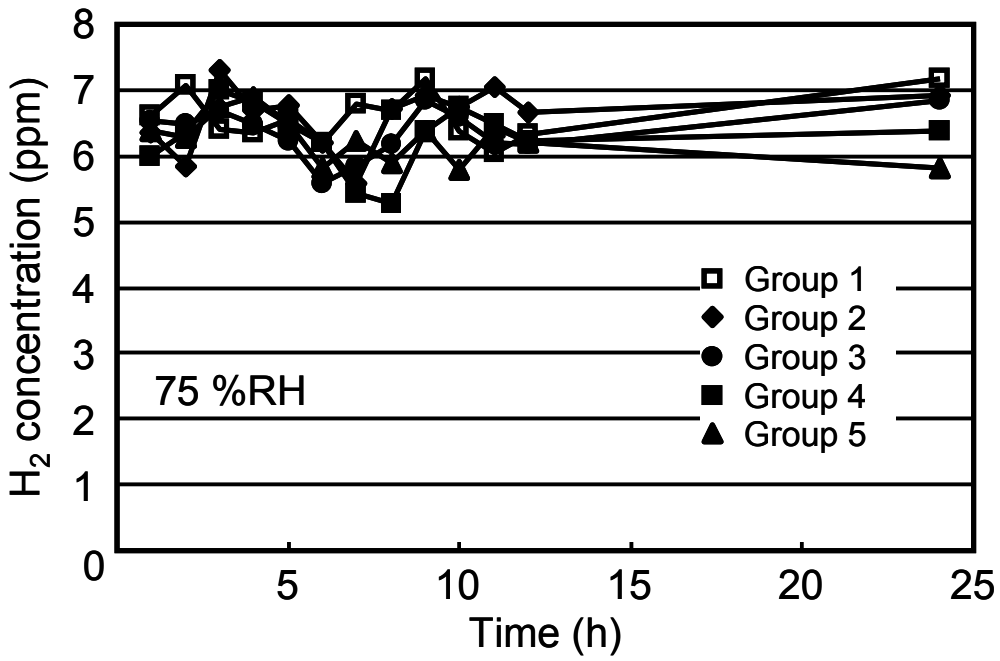

Fig. 5.8. Hydrogen concentration of at 28°C and 75%RH for five termite groups of 200 workers and 20 soldiers of *C. formosanus* from laboratory colony.

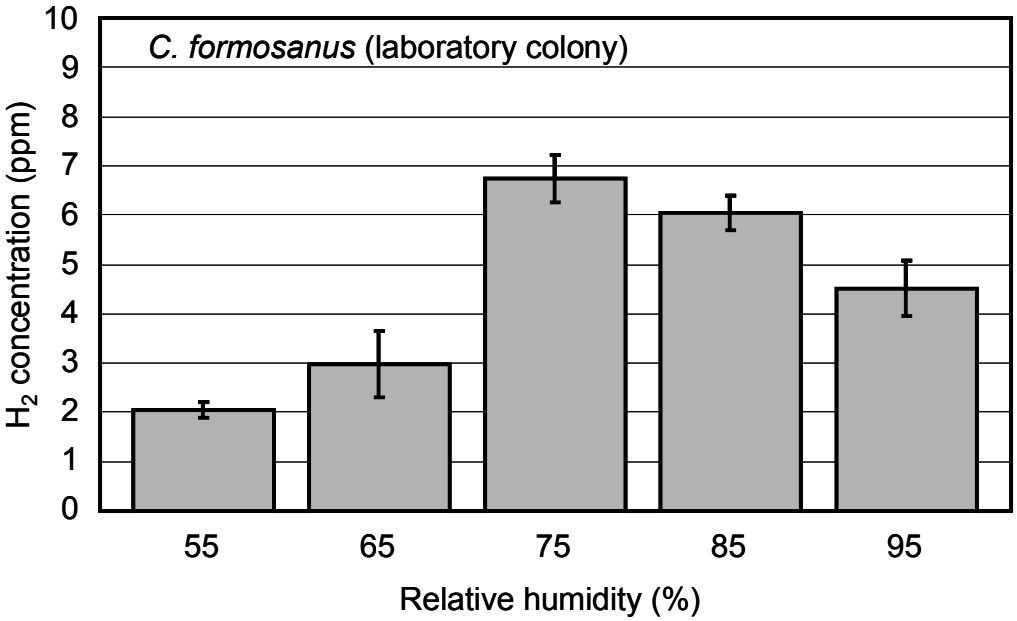

Fig. 5.9. Hydrogen concentration at five relative humidities for *C. formosanus* from laboratory colony.

The concentration was measured at 24 h from the beginning of each experiment.

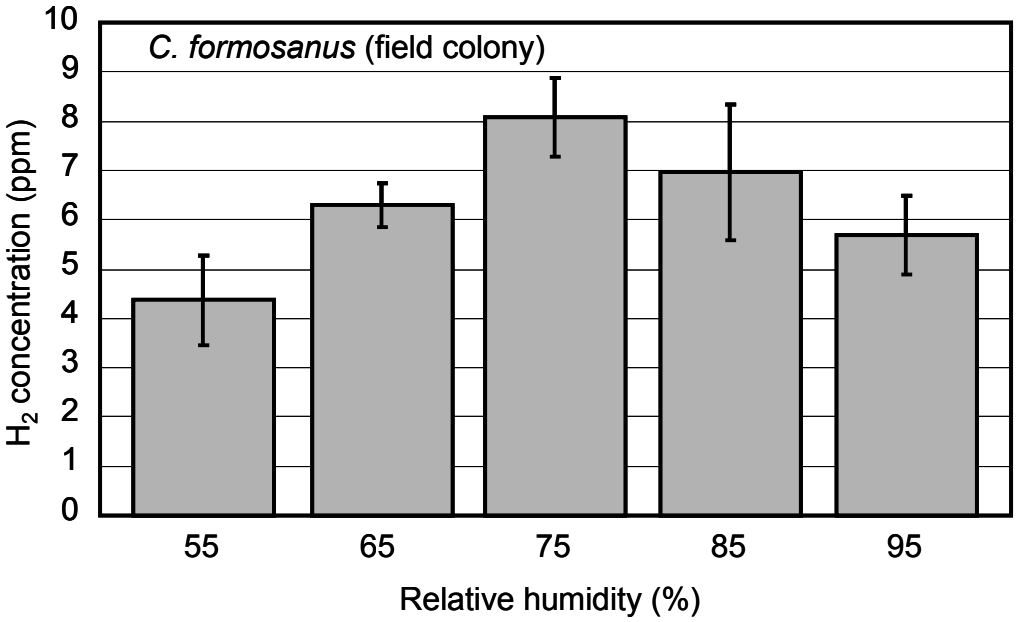

Fig. 5.10. Hydrogen concentration at five relative humidities for *C. formosanus* from field colony.

The concentration was measured at 24 h from the beginning of each experiment.

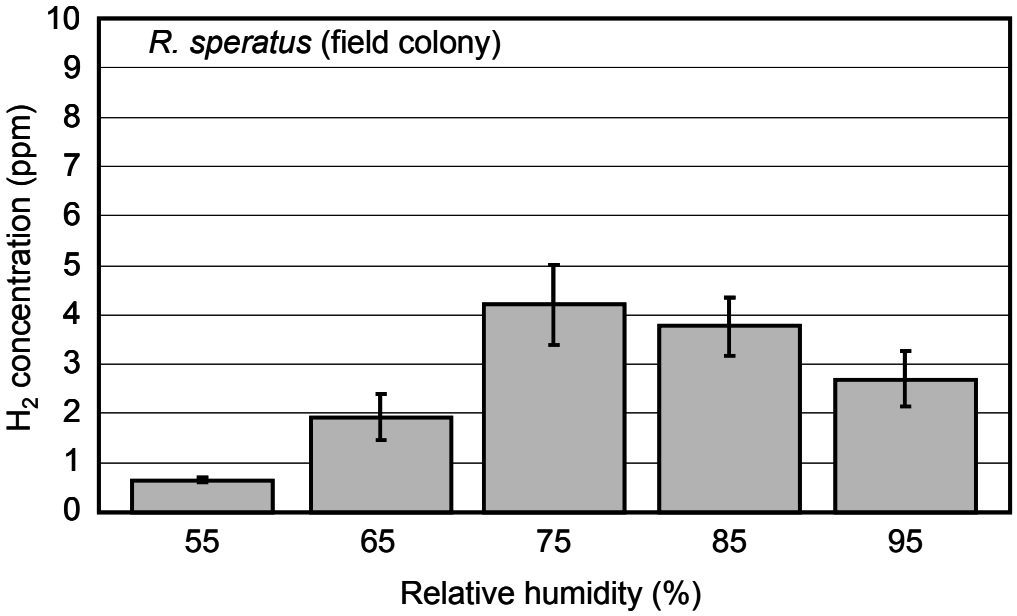

Fig. 5.11. Hydrogen concentration at five relative humidities for *R. speratus* from field colony.

The concentration was measured at 24 h from the beginning of each experiment.

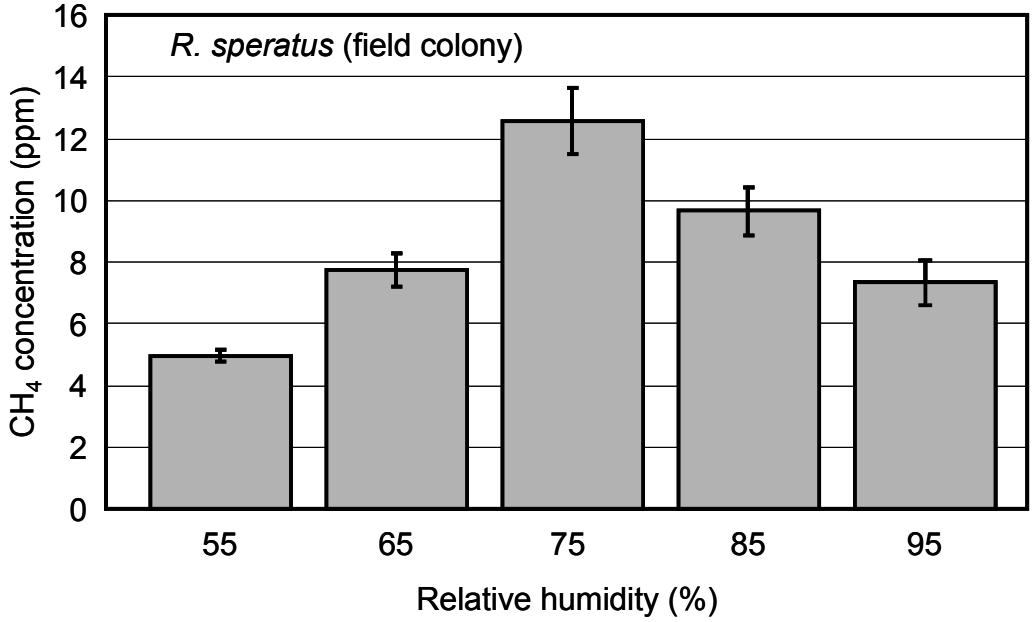

Fig. 5.12. Methane concentration at five relative humidities for *R. speratus* from field colony.

The concentration was measured at 24 h from the beginning of each experiment.

5.4 Summary

A gas analyzer equipped with a semiconductor gas sensor was employed for the qualitative and quantitative measurement of hydrogen and methane emitted by termites under forced ventilation at a rate of 25mL/h.

Termite groups of 200 workers and 20 soldiers of *C. formosanus* from laboratory and field colony and *R. speratus* from field colony were employed for the long term measurement of the hydrogen and methane concentrations. The relationship between AE and gas concentration and the effect of ambient relative humidities on the gas concentration were also examined.

The feeding of termites was active for 12 days probably because of the forced ventilation, and the hydrogen concentration fluctuated according to the existence of paper as food material, while it was not possible to detect methane. The hydrogen concentrations for *C. formosanus* increased in 4 h after inserting a paper in the bottle and then decreased. There was a tendency that the hydrogen concentration showed peaks a few hours later than those of AE event count rate. This implies that there was a delay between the feeding of termites and the hydrogen emission by termites due to digestion of wood in their guts. For all the termite groups of *C. formosanus* and *R. speratus*, the hydrogen concentration was the highest at 75%RH. Methane was successfully detected for *R. speratus*, while it was difficult to detect it for *C. formosanus*. The methane concentration for *R. speratus* was the highest at 75%RH and the lowest at 55%RH, which was the same as for hydrogen.

These findings suggest that there is a possibility to apply the gas measurement for the monitoring of termite infestation in the monitoring station where the ventilation is more or less inevitable and in the wall of wooden construction.

Conclusion

In order to develop the method for effective detection of the termite attack in wooden constructions, the performance of three devices for detection of AE generated by the wood feeding of termites and the feasibility of the semiconductor gas sensors for the detection of the metabolic gases emitted from the termite digestive system were examined in **Part I** and **Part II**, respectively.

Part I

Chapter 1

Acoustic emission (AE) monitoring using polyvinylidene fluoride (PVDF) film with 40 μm thick as a new AE transducer was applied to the nondestructive detection of termite attack in wood. Although the sensitivity of a single-layered PVDF sensor was lower than that of a PZT sensor, the sensitivity could be increased by using multilayered PVDF. It was difficult for the PVDF sensor to detect artificial AE coming over the distance more than 250 mm in the particleboard, because the artificial AE wave was strongly attenuated at the boundaries between wood particles. For the detection of AEs generated by feeding of *Coptotermes formosanus*, the cumulative AE events detected by a single-layered PVDF sensor were about one twelfth as large as those detected by a PZT sensor, while the cumulative AE events detected by a triple-layered PVDF sensor were about one fifth as large as those detected by a PZT sensor.

Chapter 2

In order to examine the feasibility of using plate-type waveguide for effective detection of AE, AEs generated by breaking pencil leads and termite attack were detected using an AE transducer attached to wood through steel plates of four different sizes and thickness and three AE transducers attached directly to wood. The larger plates were, the larger amplitudes of the artificial AEs were detected. The AE amplitudes detected by an AE transducer through a steel plate larger than 30×30mm

were greater than the amplitude of the artificial AEs detected directly by AE transducers. In detecting AEs generated by the feeding of workers of *C. formosanus*, the cumulative AE events detected by an AE transducer through a steel plate were much larger than those of AE transducers without a plate. Since AE waves are attenuated much less in a steel plate than in wood, it is more effective to attach an AE transducer to wood through a steel plate rather than directly to the wood. For the measurement of AEs generated in the studs of the wall panel and glued laminated timber, an AE transducer was attached using a needle-type waveguide penetrating through the wall covering made of plaster board and wall paper. Significant AE events more than 10 were successfully detected from studs and girth in the wall and glued laminated timber. These findings suggest that it is feasible to use a plate-type and needle-type waveguides for detection of AEs from termite attack in wood structure.

Part II

Chapter 3

With respect to the detection of metabolic gases generated by a small number of termites using three types of semiconductor gas sensors, the performance of a hydrogen sensor was found to be better than that of odor and methane sensors. Hydrogen was generated by the feeding of workers of *C. formosanus*, while soldiers that were fed by workers generated a small amount of hydrogen.

Chapter 4

A gas analyzer equipped with a semiconductor gas sensor was employed for qualitative and quantitative measurement of hydrogen and methane emitted by termites. The concentrations of hydrogen and methane increased with the number of termites of *C. formosanus*. The concentrations of both gasses were higher in bottles with a wood specimen than without it, which implies that hydrogen and methane were emitted by the feeding of worker termites. Both hydrogen and methane concentrations were the highest at 35°C and the lowest at 15 and 5°C. They were very low at 45°C because all the termites had died in a few hours. Dampwood termite (*Zootermopsis*

nevadensis) emitted relatively high levels of hydrogen and methane, whereas the drywood termite (*Incisitermes minor*) emitted no methane because of the low probability of colonization by methanogens without contact with soil.

Chapter 5

The hydrogen and methane concentrations were measured under forced ventilation at a rate of 25mL/h, and the relationship between AE and gas concentration and the effect of relative humidity on gas concentration were also examined. The termite activity remained for 12 days under this condition. In the experiment, there was a tendency that hydrogen concentration showed peaks a few hours later than those of AE event count rate. This implies that there was a delay between the feeding of termites and the hydrogen emission by termites due to digestion of wood in their guts. For all the termite groups of *C. formosanus* and *R. speratus*, the hydrogen concentration was the highest at 75%RH. Methane was successfully detected for *R. speratus*, while it was difficult to detect it for *C. formosanus*. The methane concentration for *R. speratus* was the highest at 75%RH and the lowest at 55%RH, which was the same as for hydrogen. These findings suggest that there is a possibility to apply the gas measurement for the monitoring of termite infestation in the monitoring station where the ventilation is more or less inevitable and in the wall of wooden construction.

The present study was concluded by establishing the effective methods to detect AE generated by termite attack using PVDF sensor as a new AE transducer and the optimal metal waveguides and to detect the metabolic gases of hydrogen and methane emitted by termite feeding using a semiconductor gas sensor. These methods are also possible new methods to evaluate the ecology of the wood boring insects.

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