Characteristics of Material Damage Caused by Acid Deposition in East Asia

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Material exposure experiments were performed to evaluate the relationship between air pollution and material corrosion rates based on collaboration with researchers in China, Japan, and Korea. Qualitative and quantitative atmospheric corrosion was estimated from damage caused to bronze, copper, steel, marble, cedar, cypress, and lacquer plates exposed to outdoor and indoor conditions in certain East Asian cities. The effects of atmospheric and meteorological factors on the damage to the copper plates and marble pieces were estimated using a regression analysis. The results indicated that sulfur dioxide produced the most destruction of the materials, especially in South Korea and China. In Japan, the copper plates were damaged as a result of natural conditions and sea salt. Copper was also found to be damaged by the surface deposition of sulfur and chlorine. Meanwhile, marble was substantially degraded by gaseous sulfur dioxide, yet sulfate ions in rain had no effect. Accordingly, the analysis of air pollution from the perspective of material damage was determined to be very useful in evaluating and substantiating the intensity of air pollution in East Asia.

Key words : East Asia, air pollution, material damage, exposure test

1. Introduction

Air pollution has damaging affects on both human health and cultural landmarks. East Asian countries have a very long cultural history, and thus have numerous cultural sites and artifacts that need to be maintained for future generations. However, recent air pollution resulting from the rapid industrialization and urbanization over the past few decades in East Asia, including China, Japan, and Korea, has resulted in corrosion and damage of these cultural sites. Cultural sites are composed of a variety of materials, such as wood, paper, metal, and stone, however, due to their special position as cultural assets, collecting samples that can involve their destruction is impermissible, even

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for cultural preservation activities. With the exception of intentional destruction by humans, the environment surrounding cultural sites and artifacts would appear to be the most fundamental factor when considering their protection and preservation[1]. Various previous investigations have been carried out to assess the effects of acid rain or acidic air pollution on plant growth and human health[2~5] and the reduction of acidic pollutants. Plus an international collaborative program was already started to evaluate the effects of airborne acidifying pollutants on the corrosion of materials in September 1987, which involved exposure tests at 39 sites in 12 European countries, the United States, and Canada[6~8]. In Far East Asia, a damage investigation committee focusing on material damage to cultural sites due to air pollution was initiated in June 1993 to carry out the same kind of exposure test program as the international collaborative program mentioned above. The Far East Asian project was organized by the Air

Pollution Subcommittee of the Japanese Atmospheric Environment Society and funded by the Global Environment Research Fund of the Japanese Environment Agency. During the investigations over the past 6 years, a number of air pollution episodes have been identified resulting from sulfur dioxide emitted from coal combustion in large cities in China and other developing countries[9~11]. This type of air pollution can cause serious damage to cultural and historical artifacts and structures. A few basic studies have already been carried out to evaluate the damage suffered by cultural artifacts and sites in various cities of developing countries, such as China and Vietnam. As shown in Table 1, fog is more acidic than rain. Dew and frost are more basic than rain, although their acidity is deeply affected by the acidity of gaseous air pollutants[12]. Even for one rainfall event, the pH and conductivity can change depending on the amount of rain. As shown in Figure 1, in Japan, the pH of initial rain is usually lower than that of subsequent rain, yet the reverse has been observed in China and Vietnam. This observation suggests that cleaner rain washes the surface of materials in Japan, whereas more acidic rain remains on the surface of materials in China and Vietnam. Therefore, material damage should be evaluated for many kinds of materials, such as metal, marble, wood, and lacquer under different climate conditions. In the current study, test pieces were exposed to outdoor and indoor air at 23 sites in China, South Korea, and Japan to investigate the relationship between air pollution and material corrosion in East Asia.
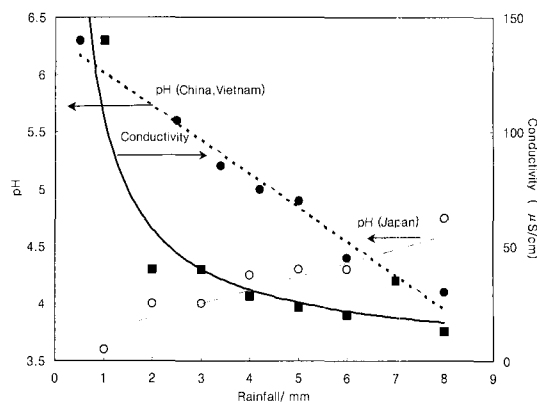

Fig. 1. pH and conductivity of each mm of rainfall.

2. Experimental

2.1 Materials

The acetone and other chemicals of reagent grade were supplied from Wako Co.(Osaka, Japan). The test pieces used in the current study were bronze, copper, steel, marble, cedar, cypress, and lacquer. The test pieces were separately exposed to both dry and wet depositions under unsheltered conditions(outdoor) and dry deposition rain- sheltered conditions(indoor). Before the exposure, the metallic plates were cleaned with acetone and the marble pieces with distilled water in an ultrasonic bath for a few minutes. The test pieces were then dried in a desiccator for 24 hours and weighed. The wooden pieces were dried at 25 ℃ in a relative humidity of 50 % for 3 days, then weighed. The lacquer pieces were prepared according to traditional Japanese methods[10].

Table 1. Acidity and chemical composition of acidic precipitation

Sampling site	N	NH_4^+	Na^+	Cl^-	NO_2^-	NO_3^-	SO_4^{2-}	CH_3COO^-	pH
Rain/Sakai	12	63.1	22.6	42.2	0.14	68.7	40.5	1.54	5.01
Rain/China	12	80.4	23.8	20.0	ND	55.8	602.3	1.85	4.61
Fog/Ikoma	10	495.6	85.5	328.8	21.7	243.6	129.4	24.5	3.82
Dew/Sakai	39	18.6	8.06	9.81	5.71	1.75	2.51	3.36	6.54
Frost/Sakai	6	19.6	10.7	11.5	2.72	2.87	2.58	0.07	7.06

Ion concentration(dew and frost : μ mol/m^2, fog and rain: μ M),
N : number of samples, ND : not detected

2.2 Apparatus

A panel prepared by Nippon Test Panel Osaka (Osaka, Japan) was used for exposure to outdoor air under unsheltered conditions and indoor air under rain-sheltered conditions. A diffusion passive sampler was used to monitor the concentration of SO_2 and NO_2 during a one-month period. A cotton gauze collector made by Nippon Test Panel Osaka was used to trap the acidic pollutants, such as acid fog, acid dew, acidic dust, and sea salt. An Elionix (Tokyo, Japan) ERAX-3000 scanning electron microscope(20 kV, 2,000 times magnifying power) was employed to assess the state of the corrosion products on the samples. A Suga(Tokyo, Japan) UGV-5D gloss meter(reflex angle at 60 degrees) and Keyence(Osaka, Japan) VH-6200 digital microscope(100 times magnification) were employed to examine the surface state of the material plates. A Rigaku(Tokyo, Japan) RINT1500 X-ray diffractometer(operating at 40 kV and 150 mA with Cuka radiation) was employed for the qualitative identification of the corrosion products. The sulfur and chlorine contents in the products were determined using a Rigaku RIX3000 fluorescence X-ray spectrometer. A Nihondenshi (Tokyo, Japan) JEOL 700 with an IR-MAU200 microscopic infrared spectrophotometer(25 μm diameter range) was utilized for the qualitative identification of the products in spots on the lacquer surface. The 7 types of test pieces were exposed to dry and wet deposits in outdoor air as well as dry deposits in indoor air. The test pieces were exposed for 1-7 years from 1993 to assess the regional effects at individual sites, and collected every 3 months to measure the seasonal impact of acid deposition.

2.3 Sampling sites

The locations of the sampling sites in East Asia are shown in Figure 2. A total of 18 sampling sites were used in the current experiment, including 6 sites in China, 10 sites in Japan, and 2 sites in Korea. The sites were classified into 6 categories as follows; heavy polluted sites in China - Chongqing and Quiyang, the urban sites in China - Beijing and Shanghai, urban sites in Korea - Taejeon and Taegu, sea coast sites in Japan - Ichikawa, urban sites in Japan - Tokyo and Osaka, and rural sites in Japan - Nara and Ibaraki. The

test pieces used in the experiment were bronze, copper, carbon steel, marble, and lacquer, which were exposed to both dry and wet depositions under unsheltered conditions(outdoor) or dry deposition rain-sheltered conditions(indoor).
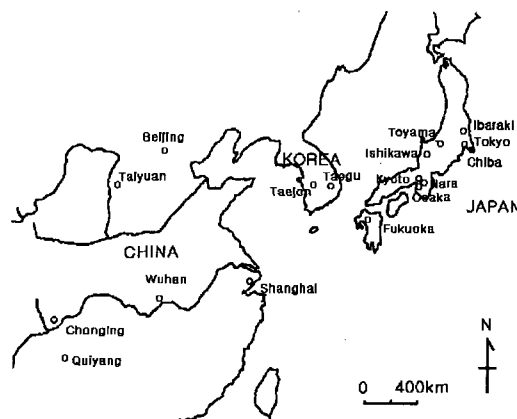

Fig. 2. The locations of sampling sites of materials damage experiments pieces.

2.4 Monitoring of environmental conditions

The test pieces were collected every exposure 3 months, 1 year, 2 years, and 4 years. Even though a variety of experiments were conducted related to the characteristics of corrosion and damage, including a chamber experiment[10], the current paper presents an analysis of the corrosion characteristics of the cultural materials. The physical properties of the metallic test pieces and marble pieces for the exposure test are shown in Table 2. The metallic pieces were ultrasonically cleaned using acetone, while the marble pieces were cleaned with distilled water for a few minutes. The test pieces were then dried in a desiccator for 24 hours, and weighed with an accuracy of less than 0.1mg. Meteorological factors, such as temperature, relative humidity(RH), and wind velocity(WV), were collected from the AWS in each country. The SO_2 and NO_2 concentrations in the ambient air and chemical composition of the precipitation were also monitored. Long- term(one month) diffusive passive samplers(DPSs) were used to measure the SO_2 and NO_2 concentrations. Bulk sampling of the acidic wet deposition was also carried out every month. The focus of the wet analysis was the pH, electrical conductivity(EC), SO_4^{2-}, NO^{3-}, Cl^-, Na^+, NH_4^+, K^+,

Table 2. The physical properties of test pieces used in this work

Materials	Exposure	Size(mm)	Component[a]	Surface polishing
Bronze	Outdoor[b]	2.0 x 30 x 40	JIS-H-5111[d]	#400 treatment
	Indoor[c]			
Copper	Outdoor	0.4 x 30 x 40	JIS-H-3100[e]	#400 treatment
	Indoor			
Steel	Outdoor	1.2 x 30 x 40	JIS-G-3141 SPPC	#400 treatment
	Indoor			
Marble	Outdoor	5 x 20 x 20	Italian white	-
	Indoor			
Cedar	Outdoor	10 x 70 x 70	Yoshino sugi	-
Cypress	Outdoor	10 x 70 x 70	Kiso hinoki	
Lacquer	Indoor	7 x 50 x 50	Glossy black lacquer	-

[a] Japanese Industrial Standard
[b] Unsheltered exposure, [c] Sheltered exposure
[d] BC6/ Cu85.6%, Sn4.9%, Pb4.9%, Zn4.6%
[e] C1201P/ Cu >99.9%

Ca^{2+}, and Mg^{2+}. The pH, EC, anions, and cations were measured using the glass electrode method, a electric conductivity meter, and ion chromatography, respectively.

2.5 Electron microscopy and X-ray diffraction analysis

The shapes and amounts of corrosion materials produced on the test pieces after exposure were observed using a scanning electron microscope. The corrosion products on the metallic plates and marble pieces were identified by an X-ray diffraction analysis.

2.6 Determination of sulfur, chlorine, and corrosion rate

The sulfur and chlorine contents in the products were determined using a fluorescence X-ray apparatus. The bronze and copper plates were washed with a 10 % ammonium-thioglycolic-acid solution at 80 ℃ for about 10 minutes to remove the corrosion products. The steel plates were washed with a boiling solution of 10 % di-ammonium-hydrogen-citrate(DAHC). The marble pieces exposed to outdoor air were dried in a desiccator for 24 hours without any pre-treatment.

The thickness loss was defined as :

thickness loss(in μm) = 10 × [weight loss(in mg) / surface area (in cm^2)] / [specific gravity (in g/cm^3)]

The corrosion rate was defined as the thickness loss relative to the exposure period for the tested pieces.

2.7 Determination of lacquer surfaces

The gloss intensities on the lacquer surfaces were determined using a gloss meter. The gloss loss was defined as the percentage of gloss intensity after exposure relative to the one before exposure. The surface state after exposure was observed using a digital microscope. The products on the surfaces were identified using a microscopic infrared spectrophotometer.

3. Results and Discussion

3.1 Meteorological factors and environmental data

The annual average values, such as the meteorological factors, dry and wet deposition related measurement data, and material corrosion rates, are summarized in tables 3, 4, and 5. These average

values were calculated on the basis of data for 6 years from 1993 to 1999. While the temperature data showed a very similar range, the RH(relative humidity) appeared to be low in the urban sites in Japan and South Korea. The WV(wind velocity) was very low(about 1 ㎥/s) in the heavy polluted areas in China, which likely deteriorated the air quality at those sites. The SO_2 concentration at the Chinese sites appeared to be much high than at the other sites, with an average value of 102.0 ppb in Chongqing, 75.2 ppb in Beijing, and 28.7 ppb in Shanghai based on a one month average. The amounts of SO_4^{2-} and Ca^{2+} in the wet deposition were also very high in China. In contrast, a higher NO_2 concentration was found in the Japanese urban areas due to vehicle emissions. Even though the Korean sites were also located in urban areas, the SO_2 and NO_2 concentrations were comparatively low because the sample sites were located on university campuses.

3.2 Microscopic observation

As shown in Figure 3, the scanning electron microscopic(SEM) images of the sample pieces

exposed to indoor air for 2 years revealed the formation of fine crystals of cuprites on the copper plate exposed in Ishikawa, Japan, while green rust covered the plate exposed in Chongqing, China. No changes were observed on the piece of marble exposed in Ibaragi, Japan, while fine crystals like bamboo blades were produced on the marble pieces exposed in Chongqing China. The surfaces of the cedar and cypress plates exposed to outdoor air, turned black with numerous fine spots(0.2 mm diameter). Numerous fine spots(0.2~0.3 mm diameter) were observed on the surface of the lacquer plates exposed in Chongqing, China and Taejon, South Korea, where dense fog occurred.

3.3 Corrosion products

The components of the corrosion on the surface of the materials observed using Electron microscopy and X-ray diffraction analysis are shown in Figure 4. The main corrosion product appeared to be $CuSO_4 \cdot 3Cu(OH)_2$ (Brochantite) in China, and $CuNO_3 \cdot 3Cu(OH)_2$ (Gerhardtite) in the Japanese urban areas. Both of these products were also identified at the Korean sites[10,11]. The formation

Table 3. Average value of the meteorological factors in East Asia from 1993 to 1999

Sampling Site		Period	Temp. (℃)	RH (%)	WV (m/s)	Rainfall (mm)	pH	SO₂ (ppb)	NO₂ (ppb)
China	Beijing	93/06/01 ~ 96/06/01	-	-	-	1659	6.3	75.2	21.5
	Chongqing	93/06/04 ~ 99/03/01	18.5	80.9	1.4	7684	5.6	102.0	24.0
	Shanghai	93/06/01 ~ 99/11/30	17.4	75.1	2.8	7080	6.5	28.7	25.6
Japan	Chiba	93/06/01 ~ 99/11/30	16.8	72.3	3.5	7562	-	8.4	23.0
	Fukuoka	95/09/01 ~ 99/12/01	15.5	78.9	1.3	7523	4.8	4.5	11.4
	Ibaraki	94/06/01 ~ 99/11/30	14.7	74.1	2.3	6732	4.8	3.5	11.9
	Ishikawa	93/06/01 ~ 98/12/01	15.4	74.1	2.2	14788	4.3	4.6	11.0
	Nara	93/06/01 ~ 99/12/01	15.8	67.7	1.8	5225	4.2	4.5	21.3
	Kyoto	93/06/02 ~ 99/12/01	16.1	77.4	1.4	8888	4.9	4.1	18.6
	Osaka	93/06/01 ~ 99/12/01	17.7	65.9	2.2	8879	4.1	6.1	32.3
	Tokyo	93/06/01 ~ 99/12/01	17.3	67.4	2.6	2063	5.8	14.9	34.0
	Toyama	95/09/01 ~ 99/11/30	14.0	77.8	2.1	13106	4.4	3.2	12.8
Korea	Taegu	93/08/05 ~ 99/11/30	15	61.8	2.8	5751	4.9	12.9	19.4
	Taejeon	95/07/30 ~ 99/11/30	13.7	70.3	1.9	5550	4.3	7.7	17.2

Table 4. Average measurement data related with dry and wet air pollution in East Asia from 1993 to 1999
$(\mu g/L)$

Sampling Site		Period	SO_4^{2-}	NO_3^-	Cl^-	NH_4^+	Na^+	Ca^{2+}
China	Beijing	93/06/01 ~ 96/06/01	84.2	25.6	7.1	19.5	2.9	24.2
	Chongqing	93/06/04 ~ 99/03/01	46.7	4.5	3.2	7.1	0.9	13.6
	Shanghai	93/06/01 ~ 99/11/30	29.1	4.3	3.6	3.9	1.8	14.1
Japan	Chiba	93/06/01 ~ 99/11/30	3.6	2.0	3.0	0.7	1.7	1.6
	Fukuoka	95/09/01 ~ 99/12/01	3.9	1.2	1.7	1.4	0.9	0.3
	Ibaraki	94/06/01 ~ 99/11/30	2.6	1.9	1.3	0.5	0.6	0.4
	Ishikawa	93/06/01 ~ 98/12/01	4.5	1.4	5.7	0.4	3.4	0.4
	Nara	93/06/01 ~ 99/12/01	7.1	1.7	1.3	1.5	0.6	0.7
	Kyoto	93/06/02 ~ 99/12/01	1.4	1.0	0.6	0.3	0.3	0.3
	Osaka	93/06/01 ~ 99/12/01	6.9	2.0	1.2	1.9	0.6	0.9
	Tokyo	93/06/01 ~ 99/12/01	2.4	1.9	2.1	0.4	0.9	2.9
	Toyama	95/09/01 ~ 99/11/30	4.1	1.5	4.4	0.6	2.6	0.4
Korea	Taegu	93/08/05 ~ 99/11/30	12.9	2.4	1.0	3.9	0.8	2.7
	Taejeon	95/07/30 ~ 99/11/30	11.6	2.7	2.4	2.9	1.3	2.7

Table 5. Average value of the corrosion rates in East Asia from 1993 to 1999

Sampling Site		Period	Corrosion rates(μm/year)							
			Marble		Steel		Bronze		Copper	
			In[a]	Out[b]	In	Out	In	Out	In	Out
China	Beijing	93/06/01 ~ 96/06/01	-	0.25	3.44	28.16	0.35	0.54	0.27	0.88
	Chongqing	93/06/04 ~ 99/03/01	3.37	18.76	29.79	143.68	2.84	5.48	1.55	4.55
	Shanghai	93/06/01 ~ 99/11/30	2.87	9.17	38.24	63.60	1.19	2.07	0.69	2.02
Japan	Chiba	93/06/01 ~ 99/11/30	-	8.09	16.78	47.30	0.61	3.15	0.45	1.86
	Fukuoka	95/09/01 ~ 99/12/01	0.54	6.61	13.67	39.99	1.05	1.17	0.54	1.66
	Ibaraki	94/06/01 ~ 99/11/30	0.33	6.01	7.09	31.84	0.13	1.36	0.22	1.27
	Ishikawa	93/06/01 ~ 98/12/01	1.04	7.74	16.54	26.96	0.97	1.77	0.61	1.63
	Nara	93/06/01 ~ 99/12/01	0.70	4.66	11.24	24.29	0.60	1.41	0.40	1.17
	Kyoto	93/06/02 ~ 99/12/01	-	7.14	18.90	34.33	0.67	1.84	0.47	1.53
	Osaka	93/06/01 ~ 99/12/01	2.00	4.61	15.75	24.06	0.59	1.22	0.46	1.06
	Tokyo	93/06/01 ~ 99/12/01	-	7.14	18.90	34.33	0.67	1.84	0.47	1.53
	Toyama	95/09/01 ~ 99/11/30	0.64	9.21	28.19	35.06	1.58	1.90	1.27	1.51
Korea	Taegu	93/08/05 ~ 99/11/30	0.65	6.79	20.79	49.18	0.64	1.65	0.52	1.75
	Taejeon	95/07/30 ~ 99/11/30	0.43	4.72	9.55	26.40	0.44	0.94	1.01	1.29

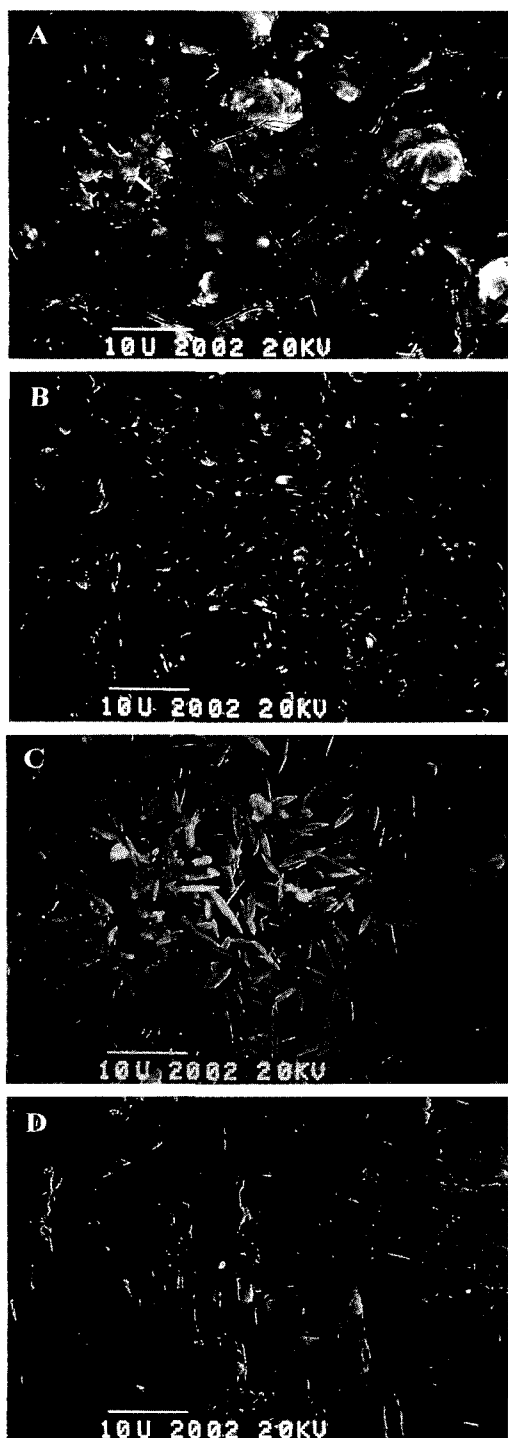[a] Sheltered exposure
[b] Unsheltered exposure

Fig. 3. The scanning electron microscopic images of the sample pieces exposed to indoor air for 2 years : Copper(A ; China, B ; Japan) and Marble(C ; China, D ; Japan) plates.

of brochantite was found to be favored at those sites where the SO_2 concentration was significantly high. Basic cupric nitrate, $Cu(NO_3)_2 \cdot 3Cu(OH)_2$, and cuprites were also detected on the copper plates exposed to indoor air in Osaka where the NO_2 concentration was relatively high and the SO_2 concentration low.
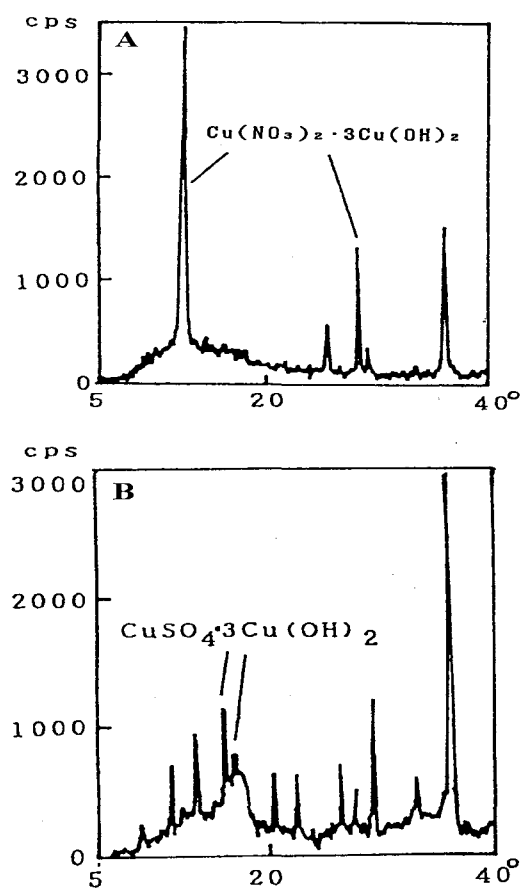
Fig. 4. The X-ray diffraction analysis of the copper plates exposed to outdoor air for 2 years : (A) Gerhardtite($Cu(NO_3)^2 \cdot 3Cu(OH)_2$) in Osaka Japan ; (B) Brochantite($CuSO_4^3 \cdot (OH)_2$) in Chongquing China.

3.4 Degree of Corrosion

The extent of corrosion was evaluated based on thickness loss in five types of test pieces. For the copper plates, the thickness loss was examined by exposing three plates every three months for one year at the same site. The results showed that the relative standard deviations of the outdoor and

indoor corrosion rates were 1.2 ~ 8.6 % and 0.9 ~ 3.4%, respectively. The thickness losses in the samples exposed to outdoor air were 2 ~ 3 times larger than those exposed to indoor air. The thickness losses for the steel plates were much higher than those for the other materials.

. The degree of corrosion decreased in the order of steel > marble > bronze > copper. Consequently, cultural artifacts and historical structures made of bronze would seem to be more sensitive to acidic air pollutants than artifacts and structures made of copper. Furthermore, copper plates(including bronze plates) may be very useful for estimating the intensity of long-term air pollution(>2 years). Figure 5 shows that the corrosion rates at the different sites decreased in the order of heavily polluted sites in China > urban sites in China > urban sites in South Korea > sea coastal sites in Japan> urban sites in Japan > rural sites in Japan. As shown in Figure 6, the corrosion of the bronze plates at all sites seemed to increase with an increase in the exposure period. The corrosion rate of the metal pieces was generally found to be proportional to the SO_2 concentration, as shown in Figure 7. The correlation coefficient between the corrosion rate of steel and the SO_2 concentration appeared to be 0.91, while that for bronze was 0.89. Conversely, the corrosion rates for the sheltered indoor marble pieces and steel exhibited different patterns, i.e. high in the urban areas and low in the heavy polluted areas in China, which would seem to indicate that the corrosion of these pieces was much more affected by wet deposition. Plus the corrosion rates of the bronze plates in Ishikawa were high comparable to those in Osaka and Tokyo. This result could indicate that the northerly winds from the Asian mainland also have an influence on the corrosion rates.

3.5 Relation between corrosion rates and atmospheric factors

. As shown in Figures 8 and 9, the corrosion rate of the materials did not decrease even when the SO_2 concentration and acidity and conductivity inthe rainwater decreased. Accordingly, air pollutants, such as SO_2, NO_X and O_3, must apparently operate synergically in order to cause significant material damage.

3.6 Copper damage and deposition flux of sulfur and chlorine

Figure 10 shows that the corrosion of the bronze plates exposed to outdoor air was related to the deposition flux of sulfur (S) and chlorine (Cl).
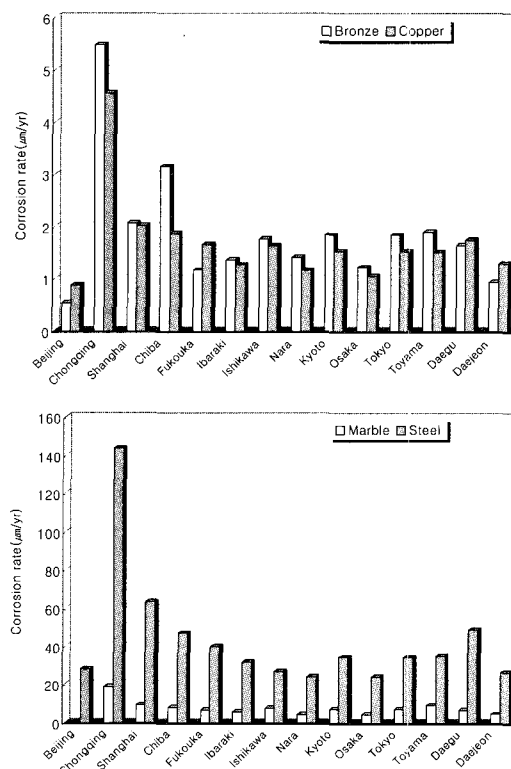

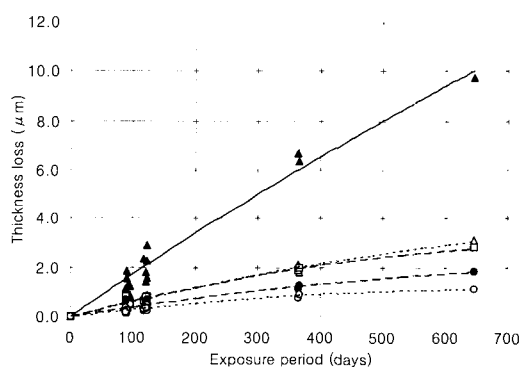Fig. 5. Corrosion rates of test pieces in outdoor.

Fig. 6. Thickness losses of bronze plates exposed to outdoor air.
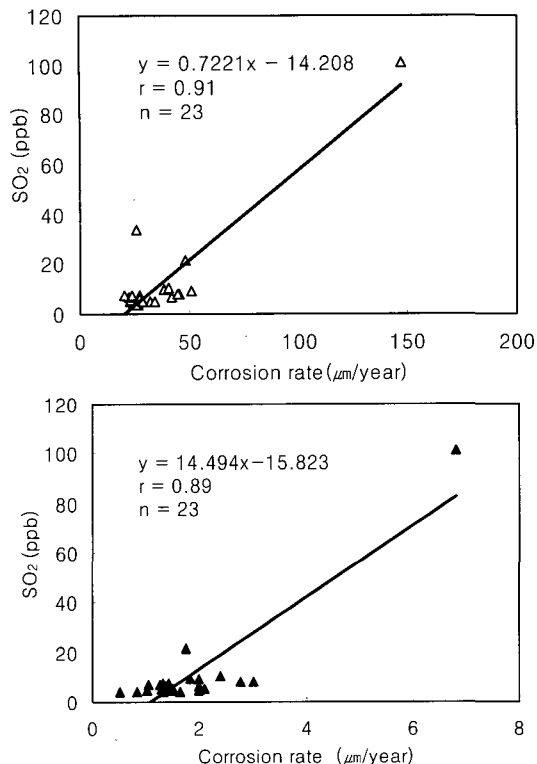▲ : Chongqing △ : Shanghai ☐ : Ishikawa
● : Osaka ○ : Kyoto

Fig. 7. The correlation between the SO_2 concentration and the corrosion rates of carbon steel ($\triangle$) and bronze($\blacktriangle$).
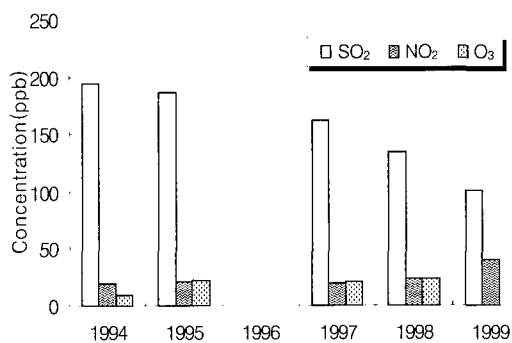

Fig. 8. Concentration of SO_2, NO_2 and O_3 in Chongqing.

The solid lines indicate the relation between the thickness loss and the deposition flux of S, while the broken lines show the relation between the thickness loss and the Cl deposition flux. At the Japanese sites, the degree of corrosion of the bronze plates increased along with the deposition flux of both S and Cl. However, at the Chinese sites, the

corrosion only increased with the S deposition flux and was unaffected by the deposition flux of Cl.
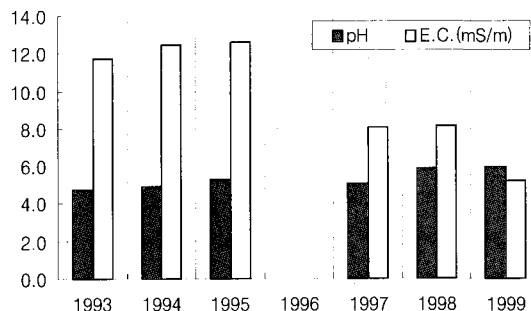

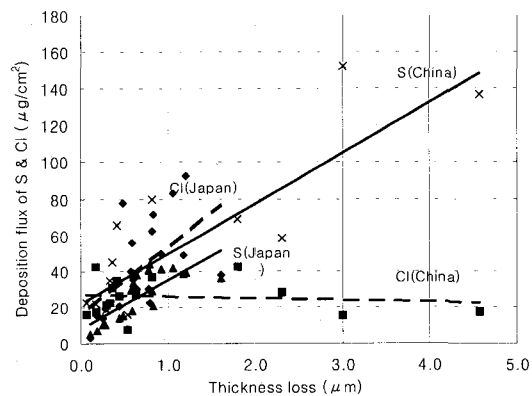Fig. 9. pH and conductivity of rain in Chongqing.

Fig. 10. The deposition flux of sulfur and chlorine on bronze plates.

4. Summary and Conclusions

Material exposure experiments were carried out to evaluate the relationship between air pollution and the material corrosion rate in collaboration with researchers in Japan, China, and Korea since 1993. As such, test materials, including bronze, copper, carbon steel, marble, cedar, cypress, and lacquer, were exposed under outdoor and indoor conditions at 18 sampling sites in East Asia. The concentrations of SO_2 and NO_2 were simultaneously measured using passive samplers. In addition, the relationship between corrosion patterns and air quality, plus the effects of natural factors, such as meteorological conditions, were also examined.

As a result, it was found that the corrosion product identified in China was mainly composed of sulfate products, while that in Japan and Korea

was mainly nitrate products. The type of corrosion found on the steel, copper, and marble pieces was characterized by type and the concentration of air pollutants. In general, the SO_2 concentration was found to be the most significant factor as regards corrosion damage to the materials. Consequently, the corrosion rate in the heavy polluted areas in China was 3~4 times larger than that in Japan and Korea. The unsheltered outdoor test pieces were also about three times more corroded than the sheltered indoor test pieces. This may have been due to wet deposition. Since damage to cultural and/or historical artifacts is very important both economically and culturally, the current regression analysis can be very useful for evaluating the intensity of air pollution in East Asia.

Acknowledgements

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