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NRCC-50804

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A version of this document is published in / Une version de ce document se trouve dans:
11th International Conference on the Durability of Building Materials and Components, Istanbul, Turkey, May 11-14, 2008, pp. 1-10
Development of an Automated Artificial Ageing Test Apparatus for Sealants and Comparison with Outdoor Exposure Testing

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ABSTRACT

The long-term performance and degree of deterioration of sealants due to ageing effects is currently evaluated using artificial accelerated test methods such as those described in JIS A 1415. However, there are many issues related to correlating the deterioration of sealants subjected to outdoor tests that are not yet resolved. In this study, a computer-controlled, automated artificial ageing test apparatus was developed that subjected sealants to a deterioration process similar to exposure outdoors. Three kinds of sealant product were selected: a 1-component modified silicone, a 2-component modified silicone, and a 1-component polyurethane, all manufactured in Japan. Outdoor environmental data was collected over one year that included, joint movement of the dynamic outdoor exposure test apparatus and specific weathering factors such as temperature of the sealant, and quantity of solar radiation and rainfall at the exposure site. The data acquired from the outdoor test was simultaneously transferred to the artificial ageing test apparatus. As a result, the movement, temperature, total radiation and wetting conditions of the artificial ageing test were nominally the same as those extracted from the outdoor test with the exception of the wavelength distribution for solar radiation. Comparing results of the deterioration of the sealant, the artificial ageing test apparatus offered a high degree of correlation to results obtained in outdoor tests for both of the modified silicone products but not for the polysulphide product. The results suggest that the development of the proposed indoor test using the automated artificial accelerated test method appears promising given the degree to which results can reproduce those obtained from ageing sealant products outdoors.

KEYWORDS

Sealant, artificial ageing, joint movement, test method, test apparatus

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1 INTRODUCTION

Factors causing the deterioration of construction sealants may be classed as, e.g., weathering, i.e. due to ultraviolet radiation, temperature and rainfall, and due to movement, that occurs by the behaviour of external walls to solar radiation [Wolf 1990; Wolf 1996]. Evidently these are the most significant factors that influence the performance of sealants over time [Wolf 1999]. Typically it is necessary to evaluate the durability of sealant products by testing before the sealant is used in building construction. At present, a dynamic outdoor exposure testing apparatus has been used at an outdoor exposure test site in Japan that provides information on the simultaneous deterioration due to weathering and movement [Enomoto et al. 2006a; Enomoto and Tanaka 2007]. However, outdoor exposure tests take considerable time to perform since the deterioration of sealants in such tests occurs in real time and from natural environmental conditions.

On the other hand, it is of evident practical importance to evaluate the durability and deterioration of sealants using tests that can be completed in a short period of time and in this context, various artificial weathering tests have previously been developed and carried out in Japan [Ito et al. 2004; Miyauchi et al. 2004]. However, being able to provide verification that results derived from outdoor exposure tests reasonably correlate to those from accelerated weathering test remains a significant research issue and a challenging task and despite the fact that a number of studies have been carried out on resolving issues regarding correlation of outdoor and accelerated weathering test results [Beasley 1996; Wolf et al. 1999; Enomoto et al. 2006b].

Various studies have been carried out to investigate the durability of sealed joints against joint movement of external walls in Japan and from these fatigue tests for sealants have been developed [Matsumoto et al. 1981; Koike et al. 1983; Miyauchi and Tanaka 2004]; this highlights the importance of joint movement in the durability of sealed joints. Hence, when considering an accelerated weathering test for sealants, it is necessary to include movement in the weathering test and to match the movement conditions with those that may be derived from the outdoors. However, it is very difficult to evaluate the deterioration of sealants using an accelerated weathering test method without outdoor test data that can be used as a basis of correlation. Therefore, in was considered that for the first stage of this study, the deterioration effects used in the artificial weathering test and their rate of application would be the same as that of the outdoor test such that the deterioration of sealants subject to both tests could be compared and the results correlated. Thereafter it is supposed that the accelerated weathering test method can be used to evaluate the deterioration of sealants given the relationships derived from correlation between test methods.

Hence, for this study, a dynamic outdoor exposure apparatus incorporating an integral joint movement system was used in outdoor exposure test [JSIA 2004] and a prototype, computer-controlled, automated artificial test apparatus was developed that permitted subjecting sealant products to a deterioration process that was similar to that obtained from outdoor exposure. Using both apparatus, the condition of sealants exposed to outdoor and artificial (indoor) testing was compared by visual observation, and by observation with a light microscope.

2 OUTLINE OF TEST METHOD FOR THE DETERIORATION OF SEALANTS

2.1 Deterioration Factors and Method of Evaluation of sealants

There are several factors that cause deterioration in sealants however, of these factors, those selected as the basic test parameters for weathering included: solar radiation, temperature, moisture and the expansion–contraction brought about by joint movement. Figure 1 shows the basic notions from which was developed a method for the performance evaluation of sealants based on artificial deterioration of products combined with outdoor environmental conditioning. First of all, environmental factors causing deterioration are divided into two components: (i) weathering effects, i.e. effects such as total solar radiation, material temperature, and moisture, and (ii) movement. Both these set of factors are measured by the dynamic outdoor exposure testing apparatus, shown in Fig. 2.
Next, the data obtained from the outdoor exposure testing apparatus in respect to the test parameters are input to instruments on the artificial test apparatus (fabricated by TiTech*) and are automatically synchronized to the outdoor conditions. The sealant surface conditions showing deterioration as characterised by both visual observation and observation by light microscope, permitted correlating results derived from either test method. Tests were carried over a 1-year period under two conditions: weathering alone and combined weathering and movement.

2.2 Test Specimen

Three kinds of sealant products were selected as shown in Table 1. The tensile testing was carried out in accordance with conditions specified in JIS K 6251 [2004] and the test specimens were prepared with a joint width and depth of 12 mm, conforming with conditions specified in ISO 13640 [1999]. The sealant was cured in accordance with conditions specified in JIS A 5758 [2004]. The surface of PU-1 was not painted to evaluate the deterioration condition of the sealant in the short term.

* TiTech – Tokyo Institute of Technology, Yokohama, Japan

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2.3 Dynamic Outdoor Exposure Test

2.4.1 Dynamic Outdoor Exposure Testing Apparatus
The dynamic outdoor exposure testing apparatus, shown in Figure 2, is able to evaluate the influence of weathering and joint movement on sealants. It is composed of a steel frame of white colour and a black coloured acrylic board. The joint movement occurs by virtue of the difference between the coefficients of linear thermal expansion of the steel frame as compared to the acrylic board.

2.4.3 Method of Measuring Outdoor Environmental Data
Total horizontal solar radiation was measured by installing a pyranometer (wavelength: 305-2800 nm) in front of the test apparatus. The surface temperature of the sealant was measured with a thermocouple embedded in the sealant 1-mm from the surface. Joint movement of test specimen was measured and calculated as follows: (i) The joint movement between the fixed and the movable structural member of the test apparatus was measured with a displacement sensor; (ii) The movement of the test specimen was calculated considering the measured value of displacement and the length of an acrylic board. The displacement sensor is protected with a metal cover to avoid the effects of solar radiation and rainfall. The presence of rainfall was measured with the rainfall sensor that made the principle of the difference of the electrical resistance between metallic needles. The amount of ultraviolet radiation was measured with a portable type UV dosimeter (UV-A type sensor).

3 OVERVIEW OF AUTOMATED ARTIFICIAL AGEING TEST APPARATUS

3.1 Method to Deteriorate Sealants
The automated artificial ageing test apparatus is shown in Figure 3 and in Figure 4, the method for installing test specimens and the measurement-control method for the test apparatus is provided. This test apparatus operates by a purposely designed control program, that is synchronized to the dynamic outdoor exposure testing apparatus.

Solar radiation in the apparatus was recreated using three sources of radiation each providing for different wavelength spectrums; the ultraviolet region was re-created using a black light, the visible region with a fluorescent lamp, and the infrared region re-created with a high-intensity filament lamp. The spectral irradiance of sunlight was used as reference. Given that the spectral irradiance differs according to specific weather conditions, the amount of irradiation from the artificial light sources was controlled by measuring the total solar radiation obtained from the pyranometer attached to the dynamic outdoor exposure testing apparatus. The quantity of total solar radiation for each weather condition was re-created by changing the number of operable sources of artificial light. The temperature condition within the apparatus was based on the surface temperature of the sealant in the

![Figure 3. Artificial weathering and movement apparatus (Side view)](image-url)
outdoor test apparatus. A heating-cooling system was used to maintain the specified temperature in the test chamber. Distilled water contained atop the apparatus in a water tank was the source of moisture used to wet the sealant surface; water was admitted via a solenoid valve through a silicon tube to the surface of the sealant as required to provide the wet conditions. A servomotor and the movable plate connected to the motor controlled joint movement. Joint movement was measured with a displacement sensor installed on the moveable plate; monitoring the movement permitted synchronization of joint movement to that of the dynamic outdoor exposure testing apparatus.

3.2 Testing Period and Condition
The testing period was one year (May 18, 2005 - May 17, 2006) and the deterioration in the surface condition of the sealant was investigated after 3, 6 and 12 months exposure. Test specimens were exposed to two (2) types of deterioration effects: weathering and the combined effects of weathering and movement. The temperature condition when initiating the test influences the behaviour of the expansion-contraction joint movement after the test. In this research, the temperature at the reference position (0 mm) was 20 °C, and the test started when the temperature was 20 °C on May 18th at 17:00 hrs. The data from both the outdoor and artificial deterioration tests were recorded every ten minutes.

3.3 Measurement Result of Outdoor Exposure Test
Figure 5 shows the results of measurement of relevant climate data and joint movement for a one year period using the dynamic outdoor exposure testing apparatus. The quantity of total solar radiation is relatively high from spring to summer, reaching a maximum of 0.97kW/m² on the 25th and 27th July, 2005. The highest sealant surface temperature was 61.4 °C on 27 July, 2005 and the lowest surface temperature was -6.6 °C obtained on 24 January, 2006. The maximum difference in sealant surface temperature over one year was 68.0 °C. In regard to the moisture condition on the sealant surface, considerable amounts of rain fell over the rainy and the typhoon season. The maximum value of joint movement in compression was -4.35 mm on 27 July, 2005 and the minimum value in expansion was 2.12 mm, occurring on 24 January, 2006. These results indicated that there was direct correlation between the temperature and joint movement; the higher the temperature, the smaller the joint opening. Given that the joint movement for the “zero-point” was calibrated at a temperature of 20 °C, the degree of movement in compression was larger than that in extension.
4 REPRODUCIBILITY OF DETERIORATION EFFECTS OF TEST APPARATUS

Figure 6 provides a comparison between results of test conditions obtained from the automated artificial ageing test apparatus (indoor) and the dynamic outdoor exposure testing apparatus. Comparative information over the test period (11 to 17 August, 2005) is provided in regard to total solar radiation (kW•m⁻²), ultraviolet radiation (W•m⁻²), sealant surface temperature (°C), surface moisture condition (rain or dry), and joint movement (mm). It can be observed that the amount of ultraviolet radiation from the artificial ageing test apparatus is smaller than that derived from the outdoor exposure test and consequently, the UV-radiation spectrum from the artificial test does not completely recreate that of the outdoors.

![Diagram showing comparison of artificial weathering test and dynamic outdoor exposure test](image-url)
5 COMPARISON OF SEALANT DETERIORATION FROM BOTH TESTS

5.1 Method of Observation for Surface Cracks on Sealants

The presence of surface cracks on sealants was investigated by two methods: that observed by using a digital camera (visual) and that with the use of an optical microscope having an attached digital scanning instrument. The detection of the presence or absence of cracks on the surface of the sealant with the optical microscope was determined by visual observation with the same magnification. The differentiation between areas having or not having cracks was determined using a light-contrasting binarization technique in which areas containing cracks appeared black and those not having cracks white in colour.

5.2 Result of Observations of Surface Cracks of Sealants

Table 2 shows the sealant crack condition for the three (3) sealants investigated (i.e. MS-1; MS-2; PU-1), the upper most set of digital photographs for MS-1, and those of sealants MS-2 and PU-1, characterised by binary imaging, provided in the lower two sets of images. The images in all three sets are those taken of the surface of the respective sealant products following exposure to outdoor testing and the corresponding artificial ageing test (indoor) for three (3), six (6) or twelve (12) months. The test results are arranged such that images can be compared between products having been subjected to only “weathering” or with “weathering and joint movement” combined.

<table>
<thead>
<tr>
<th>Sealant</th>
<th>Term (month)</th>
<th>Weathering</th>
<th>Weathering + Movement</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Outdoor</td>
<td>Indoor</td>
</tr>
<tr>
<td>MS-1</td>
<td>12</td>
<td>No crack</td>
<td>No crack</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>No crack</td>
<td>No crack</td>
</tr>
<tr>
<td>MS-2</td>
<td>6</td>
<td>No crack</td>
<td>No crack</td>
</tr>
<tr>
<td>0.5mm</td>
<td>12</td>
<td>No crack</td>
<td>No crack</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>No crack</td>
<td>No crack</td>
</tr>
<tr>
<td>0.1</td>
<td></td>
<td>No crack</td>
<td>No crack</td>
</tr>
<tr>
<td>0.5mm</td>
<td>12</td>
<td>No crack</td>
<td>No crack</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>No crack</td>
<td>No crack</td>
</tr>
<tr>
<td>PU-1</td>
<td>6</td>
<td>No crack</td>
<td>No crack</td>
</tr>
<tr>
<td>0.5mm</td>
<td>12</td>
<td>No crack</td>
<td>No crack</td>
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<td></td>
<td>3</td>
<td>No crack</td>
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</tbody>
</table>

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In regard to product MS-1, large cracks occurred to the same degree in the sealant specimens exposed to the “weathering and movement” conditions of both test apparatus; that is, the crack depth on the surface of sealants exposed in the outdoor test was 6.5 mm and for the artificial (indoor) ageing test, 5 mm. For product MS-2, the small cracks on the surface of sealants subjected to “weathering” conditions alone occurred with both test apparatus after 6 months exposure and only after 3 months when subjected to combined “weathering and joint movement”. However, larger crack sizes were evident for the same sealant exposed to the dynamic outdoor exposure testing procedure as compared that of exposure to the artificial (indoor) ageing test. For product PU-1, the surface cracks occurred after 3 months of dynamic outdoor exposure testing, whereas this occurred after 1 year for the same sealant subjected to the artificial ageing test.

5.3 Correlation of Dynamic Outdoor Exposure Testing and Artificial Ageing Test
The degree of surface crack formation of sealants subjected to the combined action of “weathering and movement” occurs earlier than those subjected to “weathering” alone. Hence the results of tests on MS-1 suggest that the joint movement has a significant influence on the occurrence of surface cracks of this product. Additionally, and most significantly, it was observed that the surface crack pattern of sealants changed from a generalised random cracking (crazing) to a series of parallel cracks by the influence of joint movement.

From a comparison of results of the sealant crack condition derived from the two test methods, it appears that the degree of factors causing deterioration in the artificial ageing test is slightly less important than that achieved with the dynamic outdoor exposure testing apparatus. It was surmised that the reason for the differences between test methods were most likely due to the influence of ultraviolet rays, specifically, the difference in UV-spectrum between outdoor and artificial ageing methods. However, other factors might also be considered to influence ageing results; e.g., the harmful effects of gas and that of acid rain. In the future it will be useful to further examine the influence of different factors causing deterioration in sealants by combining the lesser effects with the effect of UV radiation.

Additionally it would be useful to test different types of products not as susceptible to deterioration from joint movement given that for product MS-1, the influence of joint movement on the formation of cracks was clearly evident. However, it was not easy to discern differences in the degree of deterioration achieved using either test method on the basis of results of tests on this product. Nonetheless, the results suggest that the test method that incorporates both “weathering and joint movement” best recreates outdoor exposure and hence most closely replicates natural exposure conditions.

6 CONCLUSIONS
The following conclusions were obtained.
(1) The sun light spectrum of the artificial ageing test differs from the dynamic outdoor exposure test, the difference, in particular, being the ultraviolet radiation spectrum of the artificial ageing test. However, the total solar radiation, sealant surface temperature and moisture condition from the outdoor environmental conditions were readily replicated in the artificial ageing test apparatus.
(2) The surface crack condition for the PU-1 sealant product when subjected to the artificial ageing test was different than that of the same product exposed to the dynamic outdoor exposure test, however, the surface crack condition of sealant products MS-1 and MS-2 resulted in nominally the same conditions from the artificial ageing as compared to that of the outdoor exposure test.
(3) The degree of crack propagation in similar sealant products occurs earlier in those sealants subjected to the combined action of weathering and joint movement as compared to those subjected to weathering alone.
ACKNOWLEDGEMENTS

The authors would like to express their sincere gratitude to committee members of the JAPAN SEALANT INDUSTRY ASSOCIATION and to Mr. Duong Minh Nhut and Mr. Wataru Hirasawa.

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