Abstract

Anti-carbonation coatings for concrete act as barriers to carbon dioxide and thus prevent carbonation. They are extensively used in the rehabilitation of concrete structures throughout the world to prolong the service life of a structure. This paper describes a project, which aims to evaluate the performance of coatings after natural and artificial weathering. Test methods currently available in draft and current CEN Standards (crack bridging, water vapour resistance, CO₂ diffusion resistance and liquid water resistance) will be used. The duration of the programme will be five years for natural weathering and a minimum of 2500 hours QUV exposure for artificial weathering.

The project started in April 1997 and should be completed in March 2003. Ten different coating systems have been applied to different strength concrete beams, mortar specimens and unglazed ceramic tiles. Urban and marine sites are being used to naturally weather the specimens. The coatings will be monitored during the period of weathering to investigate changes in the physical properties of the coating. From this information a classification system will be evolved to guide the specifier in the selection of a coating to suit their purpose. The work will also be used to validate European Standard tests and classification systems currently being drafted. This paper summarises the work to-date and background information on the project.

Keywords: coatings, concrete, carbon dioxide, resistance, diffusion, permeability, water vapour
1 Introduction

Problems associated with carbonation of concrete have been thoroughly documented (Building Research Establishment 1982, Everett, L.H. et al, 1980, Concrete Society, London 1984, Currie, R.J 1986, Rothwell G.W. 1985). Carbonation may result in corrosion of depassivated reinforcement, and deterioration and loss of serviceability of structures. Surface coatings and treatments are increasingly considered as an integral part of the repair and maintenance process to reduce the rate of further deterioration (CIRIA Technical Note 130, 1987).

Surface treatments to protect concrete must have certain key of properties to be effective and durable. Anti-carbonation coatings are surface treatments that have low permeability to carbon dioxide and reduce the risk of carbonation. Typical properties that a surface treatment needs to prevent carbonation are:

- a dry film thickness of at least 200 μm, in order to cover effectively a rough concrete surface and a defect free film
- good adherence
- ability to accommodate anticipated movements in concrete; this is specified as the crack-bridging ability of a coating.
- good colour retention, chalking and ultra-violet (UV) resistance
- a high liquid water resistance
- an low water vapour resistance
- a high resistance to carbon dioxide
- a service life of at least 10 years

In future the selection of coatings for concrete in Europe will be specified by European Standards. The aim of this project is to evaluate the performance of anti-carbonation coatings and to see whether the test methods, proposed classes and classification system in the European standards are appropriate for selecting anti-carbonation coatings.

2 European standards and future selection of coatings for concrete

A fundamental goal of the European Union (EU) is to remove barriers to trade among member states. Replacement of national standards by European Standards is a key element in the single market policy. All members of the EU have signed the Construction Products Directive (CPD), which has led to the development of European Standards for the construction industry. In the next two to three years, national standards in member states of the EU for surface treatment of concrete will be replaced by European Standards.

These developments have serious implications for UK industry’s approach to standards. It is important to realise that the Single Market requires a culture change in the way standards are used. An important consequence of European Standards is that public bodies are required to specify wherever possible to European standards for all construction products and this include coatings for concrete, i.e. European Standards are de-facto mandatory for public or privatised utilities if applicable.
Most of the European Standards and Classification Systems for coatings on concrete will be in force within the next three years. Of particular interest to the industry will be draft EN 1062-1 (Classification for Masonry Paints), draft EN 1504-2 (Classification System for Surface Treatment of Concrete), ENV 1504-9 (Guidance note on the principles of protection and repair of concrete) and draft EN 1504-10 (Site application of products and systems and quality control of the works).

Surface treatments for concrete with a maximum dry film thickness of 5 mm are covered by draft EN 1504-2. This standard will give guidance on the selection of coatings for concrete and the required mandatory tests for properties of coatings. For example, if a coating is to be used for ingress control, the manufacturer will have to test the coating for adhesion (pull-off test) and carbon dioxide, liquid water and water vapour permeabilities.

3 Materials, specimen preparation and exposure

Ten commonly used commercial coating materials were selected. The coatings were applied to unglazed ceramic tiles and prisms of concrete (100 x 100 x 750 mm) with 20 N and 33 N strength. The ceramic tiles were selected for their high porosity, low resistance to carbon dioxide and their low relative cost. Specimens will be naturally aged on the south coast of England and at BRE for 5 years, and artificially weathered in a QUV machine for at least 2500 hours. The specimens will be removed periodically during the weathering programs and the tested for the following properties:
• Colour and gloss of coatings
• Carbon dioxide permeability of coatings
• Water vapour permeability
• Adhesion of coatings to concrete
• Carbonation depths of concrete
• Crack-bridging ability of coatings

The initial tests on the specimens i.e. time zero, will be completed by the end of November 1998, and the weathering programs will then start. The tests used in the program and their backgrounds to the test are described below.

4 Carbon dioxide permeability

Systems for measuring the carbon dioxide resistance of coatings can be classified according to the type of test cell used. There are two types available; the absorption cup (or gravimetric method) and systems based on the diffusion cell. Those based on the cup method rely on the absorption of CO₂ in the cup. Absorption is measured by weighing the cup regularly to determine the increase in weight against time. Diffusion cell systems measure the quantity of CO₂ passing through the membrane test by analysis of the gas stream.
A gravimetric test developed at BRE has shown that the test of carbonation of concrete may be reduced by a number of different coatings and surface treatments (Rothwell, G.W, et al, 1989). Further work has shown however, that coatings could become less effective carbon dioxide barriers over a period of 2000 hours artificial weathering (Wang Wei, et al, 1990), that the reduction of barrier properties varied according to the chemical type of binder used in the coating, and that the reduction was more significant for some coatings than for others.

Klopfer and Engelfried (Engelfried, R, 1977, Engelfried, R, 1983) also developed a gravimetric method of measuring resistance of coatings to carbon dioxide. They tested resistance on paper substrates, which are not suitable either for natural or for artificial weathering. The gravimetric method developed at BRE uses cementitious substrates and is destructive. It has limited sensitivity and is not suitable for measuring carbon dioxide resistance of high performance coatings. To overcome the limitations of the gravimetric test a non-destructive method of test using an isostatic partition cell has been developed.

The changes in performance of a coating produced by accelerated weathering need to be confirmed by measuring performance over a period of natural exposure. Natural exposure test data are also needed to determine the relationship between accelerated test results and likely in-service performance. This will allow an accelerated exposure regime for the prediction of carbon dioxide resistance of coatings to be defined.

The measurement of carbon dioxide resistance of coatings has been carried out for over 15 years in the UK, Denmark and Germany, but the methods used and the results obtained are not widely understood.

4.1 Apparatus

Development of partition cell apparatus

Thomas and Gent (Thomas, A.M., et al 1945) used a partition cell to study permeation and sorption of moisture vapour in varnish films. A similar method was employed to measure the permeability of plastic films for packaging applications (Davis, W.W., 1946). Pasternak, Schimscheiner and Heller (Pasternak, R.A. et al, 1970) first proposed the use of a partition cell coupled with a gas chromatograph to determine the permeability of films to gas mixtures, and this was further developed by Pye, H. Ehnh and Panar (Pye, D.G., et al, 1976).

Several others have developed apparatus based on the partition cell. Möller (Möller, L., 1985) used a partition cell with gravimetric determination derived from Sherwood’s report. Others employed instrumental techniques to measure the rate of diffusion of carbon dioxide through the test sample. Désor and Pauly (Désor, U. et al, 1985, Désor, U., 1985) used infrared analysis to determine the rate of carbon dioxide diffusion through coatings. Robinson (Robinson, H., 1986, Robinson, H., 1987) used a gas chromatograph to measure diffusion rates of oxygen and carbon dioxide using dry gas mixtures. The equipment employed by these three is all derived from that reported by Davis for testing the carbon dioxide resistance of packaging materials (Davis, W.W., 1946). The present study was carried out using a similar system of partition cells and gas chromatographic analysis. The method of analysis by Durbeck and Niehaus (Durbeck, H.W et al, 1978), Gates et al (Gates, W., et al, 1981), Kern and Elser (Kern, H., et al, 1978) and Latif et al (Latif, S., et al, 1983). These data indicated that mixtures of gases containing water vapour could be analysed successfully by gas chromatography.

4.2 BRE Carbon dioxide permeability test

The BRE test methods is based on a partition cell see Figure 1. The apparatus has three main parts (Figure 2). A gas handling system provides a stream of a mixed, dried test gas and a helium stream, which are linked to opposite sides of a differential mercury manometer. The pressure in the two streams is equalised. The test stream is supplied through a manifold to the lower chamber of each cell, where it may be humidified. The test gas is a mixture of 15% carbon dioxide in nitrogen. Helium is supplied at a pre-set flow rate to the upper chamber of each cell via a similar manifold. The manifolds also act as ballast volumes to smooth minor fluctuations in the flow rates through the cells.

The coating under test is supported on a porous ceramic tile to form the test membrane. A plastic ring clamps the membrane against an ‘O’ ring mounted in a rebate in the wall of the upper chamber, forming a seal and defining the area of coating under test. The pressure balance ensures that the two chambers of each cell contain different partial pressures of gases at the same overall pressure.

Gas transport through the membrane occurs by diffusion. Gas diffusing through the membrane from the lower (test gas) chamber enters the sweep-stream in the upper chamber. At equilibrium the rates of diffusion of the component gases in the test mixture and of helium through the membrane reach a steady state and the test gas content of the helium sweep-stream reaches a constant value.

A selection valve connects the cells to the sampling valve of the gas. The sweep gas of each cell is sampled in turn followed by the helium and test streams. The cycle is repeated ten times. Typically the steady-state condition is reached in about eight hours and the data processor controlling the apparatus is programmed to analyse the gases after this period. Examination of the data after the analytical cycle is complete indicates when equilibrium was attained, and any analyses carried out before equilibration was achieved is discounted and the flux calculated for the remaining data. A plot of the GC detector response against time give a series of gaussian peaks, each of which correspond to one component of the test gas mixture under ideal conditions. A digital integrator gives values of peak area,
height and retention time. The peak area is used to determine the concentration of carbon dioxide in the samples from the partition cells.

Given the volume of sample injected, the flow rate of the sweep helium through the partition cell and the concentration of CO\(_2\) in the sample the quantity of carbon dioxide passing through the specimen per minute may be calculated, thus:

\[
Q = \frac{F \cdot C \cdot v \cdot M}{V} \times 10^{-6} \text{ g min}^{-1}
\]

where:
- \(F\) = flow rate of helium sweep stream in cm\(^3\) s\(^{-1}\)
- \(C\) = concentration of CO\(_2\) in sample
- \(v\) = sample volume in cm\(^3\)
- \(M\) = relative molecular mass of CO\(_2\)
- \(V\) = molar gas volume at 200\(^\circ\)C
- \(Q\) = quantity of CO\(_2\) in g min\(^{-1}\) passing through the coating at steady state

From this value it is possible to calculate the flux, \(J_{\text{CO2}}\), in g m\(^{-2}\) day\(^{-1}\):

\[
J_{\text{CO2}} = \frac{Q \times 1440}{A} \text{ g m}^{-2} \text{ day}^{-1}
\]

where \(A\) = area of coating in m\(^2\)

4.2.1 Measurement and calculation of carbon dioxide resistance

Carbon dioxide resistance may be calculated and quoted in several ways. The terms and units most commonly used are: flux (g m\(^{-2}\) day\(^{-1}\)); diffusion coefficient (m\(^2\) s\(^{-1}\)); diffusion resistance coefficient (dimensionless) and equivalent air layer thickness (m).

The derivation and calculation of these values is as follows: the flux is a function of the concentration gradient across the coating and of the thickness of the coating. Applying Fick’s second law of diffusion, it is possible to determine the coefficient of diffusion for a given thickness of coating, \(D_{\text{Coating}}\), from the flux and concentration difference of CO\(_2\) across the coating, thus:

\[
D_{\text{Coating}} = \frac{J_{\text{CO2}} \cdot s}{[c]}
\]

where:
- \(J_{\text{CO2}}\) = flux (g m\(^{-2}\) s\(^{-1}\))
- \(s\) = thickness (m)
- \([c]\) = concentration difference (g m\(^{-3}\))

This value has units (m\(^2\) s\(^{-1}\)), and is therefore not a dimensionless coefficient. It is converted to a dimensionless diffusion resistance coefficient, \(\mu\), which compares the diffusion of CO\(_2\) through the coating with the diffusion of CO\(_2\) through still air under the same conditions and is independent of applied coating thickness. It is calculated as follows:

\[
\mu = \frac{D_{\text{CO2}}}{D_{\text{Coating}}}
\]

where \(D_{\text{CO2}}\) = diffusion coefficient of CO\(_2\) through air (m\(^2\) s\(^{-1}\))
Typical values for the diffusion resistance coefficient are in the range 10^6 to 10^8 for surface coatings with a high resistance to CO\textsubscript{2}. This coefficient is used to calculate the effectiveness of the coating in terms of the equivalent air layer thickness, \( R_{(CO_2)} \). The equivalent air layer thickness equates the barrier property of a coating to the thickness of still air with the same diffusion resistance. It is obtained by multiplying the diffusion resistance coefficient, \( \mu \), by the coating thickness, \( s \), as follows:

\[
R_{CO_2} = \mu \cdot s
\]  

(5)

Typical values for \( R_{(CO_2)} \) are between 1 m and 500 m, but higher values are not unknown.

4.2.1 Effects of relative humidity on carbon dioxide resistance

Research at BRE (unpublished) has shown that there are some variations in the values obtained from measurements carried out at the two humidities of test gas, but the general trends observed are similar at both humidities.

The humidity at which the test is conducted does not have as much effect on the measured carbon dioxide resistance as the type of chemical binder used in the coating or the duration of exposure of the coating. In practice, coatings are applied in conditions where they will almost invariably be exposed to moist air. It would therefore see prudent, where possible, to carry out the measurement of carbon dioxide resistance using a humidified test gas.

### 4.3 Gravimetric method for measuring carbon dioxide permeability

The gravimetric method can only be carried out with humidified test gas if the humidity of the whole test apparatus is maintained at a uniform level. This may prove difficult with the gravimetric method based on the Payne Cup tests (Draft EN 1062-2, ASTM E96-80, 1980, DIN 53 122, 1974). These usually employ an absorbent solid on one side of the coating, and these absorbents are usually absorb water as well as carbon dioxide.

### 5 Water vapour permeability test

5.1 BS 3177:1959 Cup method for determining water permeability

BS 3177 employs an aluminium dish, commonly called a “Payne Cup”. The depth of the aluminium dish is dictated by the permeability of the paint system to be tested, the lower the permeability the shallower the dish. The largest dish size recommended by the standard is usually chosen, which gives a test area of 50 cm\textsuperscript{2}. Calcium chloride is used as the desiccant. A mechanical or wax seal used to seal the specimens. In the test program a mechanical seal is used, so that it is a non-destructive test. The test is usually undertaken at 23\textdegree C and 55±5 %RH.

EN 1062-1 uses a Payne cup for the determination of the permeability of paints, as specified in ISO 7782. However, in draft EN 1062-1 the humidity gradient across the coating is obtained by sealing a humidifying solution into the cup, not a desiccant. Also, the coatings are subjected to a weathering cycle prior to testing. Unpublished result at BRE has shown that the results obtained using the two methods are similar.
6 Crack-bridging test

A crack-bridging test is undertaken to determine a coating’s ability to accommodate movement of cracks in concrete due to load and temperature changes. There are two types of test, static and dynamic. The static test is a one-cycle test, which will give an indication of the maximum crack width that can be accommodated by a coating. The dynamic test will give information about the performance of a coating over an active crack.

6.1 Requirements of the draft European standard (1062-7)

6.1.1 Dynamic crack-bridging test

The latest draft requires that tests be carried out in triplicate. It does not state the design of the test specimen. The draft specifies that the mortar for the specimens for the static and dynamic crack-bridging test should be in accordance with EN 196-1 as follows:

- OPC 4.5 kg
- Water 2.25 kg
- Sand 13.5 kg

For the dynamic test the proposed crack displacement cycle should follow the movements shown in Figure 2 and the requirements for the different classes are given in Table 1. Classes B0 and B1 are for coatings with a minimal requirement for crack bridging, such as tanking coatings. Class B2.1 is for coatings on structures which may be subjected to crack movements due to thermal changes and loading, but not mechanical loading. Class B2.2 is for coatings with the same requirements as class B2.1, but subjected to mechanical loads, such as liquid applied waterproofing systems on bridges, under asphalt. Class B3.3 is for coatings with the same requirements as for class B2.2, but with a large crack movement requirement and is for coatings subjected to direct traffic as in a car park or warehouse. The test is undertaken at one of the following temperatures: 23°C, 10°C, 0°C, -10°C or -20°C. The test temperature selected should be the most appropriate to the expected service conditions. The proposed pass criterion for the dynamic crack-bridging test is that the coating should remain intact after the test.
Table 2: Draft European Standards (prEN 1062-7) test conditions for different Dynamic crack-bridging requirements for surface treatments

<table>
<thead>
<tr>
<th>Class</th>
<th>Test conditions (see Figure 1)</th>
</tr>
</thead>
</table>
| B 0   | \(w_0 = 0.15 \text{ mm}\)  
\(w_u = 0.10 \text{ mm trapezoid}\)  
\(RW = 100\)  
\(f = 0.01 \text{ Hz}\)  
\(W = 0.05 \text{ mm}\) |
| B 1   | \(w_0 = 0.15 \text{ mm}\)  
\(w_u = 0.10 \text{ mm trapezoid}\)  
\(RW = 100\)  
\(f = 0.01 \text{ Hz}\)  
\(W = 0.05 \text{ mm}\) |
| B 2.1 | \(w_0 = 0.25 \text{ mm}\)  
\(w_u = 0.10 \text{ mm trapezoid}\)  
\(RW = 1000\)  
\(f = 0.03 \text{ Hz}\)  
\(W = 0.15 \text{ mm}\) |
| B 2.2 | As above, and  
\(w_L = \forall 0.05 \text{ mm sinusoidal}\)  
\(RW = 20,000\)  
\(f = 1 \text{ Hz}\) |
| B 3.1 | \(w_0 = 0.50 \text{ mm}\)  
\(w_u = 0.20 \text{ mm trapezoid}\)  
\(RW = 1000\)  
\(f = 0.03 \text{ Hz}\)  
\(W = 0.30 \text{ mm}\) |
| B 3.2 | As above, and  
\(w_L = \forall 0.05 \text{ mm sinusoidal}\)  
\(RW = 20,000\)  
\(f = 1 \text{ Hz}\) |

Key:

\(f\) = frequency  
\(w_L\) = super imposed crack movement  
\(w_0\) = maximum crack width  
\(w_u\) = minimum crack width  
\(RW\) = number of crack cycles  
\(W\) = change in crack width

6.1.2 Static crack-bridging test

The crack displacement rate for the static crack-bridging test should be 0.1 or 0.5 mm per minute. The rate of 0.5 mm per minute is to be used when the static crack-bridging ability of coating is likely to be known to be greater than 3 mm, in order to reduce the time spent testing. The crack shall be induced in the test...
specimen at 23°C prior to testing. Four classes have been agreed for the static crack-bridging test and are listed below.

<table>
<thead>
<tr>
<th>Class</th>
<th>Temperature (°C)</th>
<th>Crack Width (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>23</td>
<td>&gt;100</td>
</tr>
<tr>
<td>2</td>
<td>-10</td>
<td>&gt;250</td>
</tr>
<tr>
<td>3</td>
<td>-10</td>
<td>&gt;1250</td>
</tr>
<tr>
<td>4</td>
<td>-10</td>
<td>&gt;2500</td>
</tr>
</tbody>
</table>

7 Conclusion

The work will also be used to validate European Standard tests and classification systems currently being drafted. This paper summarises the work to-date and background information on the project. Data on the testing of the coating will be presented at the conference.

8 References


Draft EN 1062 – 1 Masonry


Fig 2a: Change of crack width as a function of time for classes B2.2 and B3.2. 1000 crack cycles as a trapezoidal function using 0.03 Hz, with superimposed crack cycles as a sinusoidal function using 1Hz

Fig 2b: Change of crack width as a function of time for classes B0, B1, B2.1 and B3.1. 1000 crack cycles as a trapezoidal function using 0.03 Hz

**Fig. 2:** Crack width profiles for the draft European dynamic crack-bridging test
Fig. 1: Schematic diagram of partition cell