Materials and Structures/Matériaux et Constructions, 1989, **22**, 225–230

CEMBUREAU RECOMMENDATION

The determination of the permeability of concrete to oxygen by the Cembureau method – a recommendation

J. J. KOLLEK

A committee was set up in 1981 by the Cembureau Working Party on the Quality of Concrete with the following aims and objectives:

1. To develop a suitable method for the measurement of the permeability of concrete to gas or water and to determine the effects of mix proportions and curing.

2. To establish the feasibility of obtaining concordant results in a number of participating laboratories.

Eight European laboratories have participated in the study and carried out a number of co-operative programmes in the course of six years. This recommendation of the method, inclusive of apparatus and procedure, is based on the findings of the committee. The work has given rise to a number of reports, some of which have been published with the approval of this committee. A limited bibliography of recent papers which the committee has considered and discussed will be found appended to this recommendation.

The chairmen of this committee were Mr H. W. W. Pollitt (1981–85) and Mr P. Dutron (1985–87). The address of Cembureau is 55 rue d'Arlon, B-1040 Bruxelles, Belgium.

CONTENTS

1. INTRODUCTION

Recent concerns about the long-term performance of concrete led to interest being focused on parameters which control the durability and methods of test which meaningfully quantify these parameters. There is a growing realization that the cube crushing strength, for long the sole indicator of quality, is insufficient to ensure long-term durability for concrete as a material and serviceability of a structure made from it. Other material factors should be explored with a view to establishing whether meaningful additional information could be provided, and whether such information could be determined in a manner which is commensurate with the application of concrete.

Pore structure has long been recognized as strongly relating to many, if not all, aspects of durability, and it is felt that some form of permeability test might be devised which would have the necessary features. Of primary interest in a typical concrete structure is the surface region which protects the reinforcement from corrosion and which is subject to penetration by oxygen, carbon dioxide and water. The practicalities of a test dictate that the latter two fluids are cumbersome in that they themselves modify the pore structure of concrete, and as such are less suited to a repeatable and reproducible test. For this reason oxygen was selected as a permeating medium. The permeability test cell together with the sealing system was conceived as a simple, repeatable and yet efficient means of constraining the specimen to the

0025–5432/89

designed test parameters with only a remote chance of inadvertent obstruction or impairment of the specimen. The dimensions of the standard disc specimen (50 mm thick, 150 mm in diameter) were selected on consideration of the volume coinciding with that of a 100 mm cube and the selection of thickness at about twice the typical cover to reinforcement.

The pressure difference was selected on the basis that the most meaningful pressure differences for concretes are of low magnitude, but the need for a reasonably large flow rate to suit simple instrumentation dictated a compromise at between 1.5 to 3.5×10^5 N m^{-2} absolute (0.5 to 2.5 bar above atmospheric pressure).

2. SCOPE

This recommendation defines and describes the test apparatus and lays down a procedure which can reasonably be expected to give repeatable and reproducible results at practicable precision. The method is intended to be suitable for concretes of normal composition within the range of 200 to 450 kg m^{-3} of cement, giving a specific permeability coefficient K to oxygen within the range of 10^{-14} to 10^{-19} m^2. The method is applicable to cast and cored specimens within the dimensional tolerances dictated by the apparatus and as specified in Section 4 below. Deviation from either the specification of the apparatus or the laid-down procedure is unlikely to yield results directly comparable with those obtained by this method.

3. PRINCIPLE AND GOVERNING RELATIONSHIP

The underlying principle is the Hagen–Poiseuille relationship for laminar flow of a compressible fluid through a porous body with small capillaries under steady-state conditions. The relationship solved for the specific permeability coefficient K can be written

$$K = \frac{2Qp_0L\eta}{A(p^2 - p_a^2)} \quad (\mathrm{m}^2) \qquad (1)$$

where Q = volume flow rate of the fluid (m^3 s^{-1}), A = cross-sectional area of the specimen (m^2), L = thickness of the specimen in the direction of flow (m), η = dynamic viscosity of the fluid at test temperature (N s m^{-2}), p = inlet pressure (absolute) (N m^{-2}), p_a = outlet pressure assumed in this test to be equal to atmospheric pressure (N m^{-2}) and p_o = pressure at which the volume flow rate is determined, assumed in this test to be atmospheric pressure p_a (N m^{-2}).

For oxygen at a temperature of 20°C the dynamic viscosity may be taken at 2.02×10^{-5} N s m^{-2}; thus

$$K_{\mathrm{oxygen}} = \frac{4.04 \times 10^{-5} Qp_aL}{A(p^2 - p_a^2)} \quad (\mathrm{m}^2) \qquad (2)$$

and when the standard reference specimen of 150 mm diameter and 50 mm thickness is used the relationship is further simplified to

$$K_{\mathrm{oxygen}} = \frac{1.14 \times 10^{-4} Qp_a}{(p^2 - p_a^2)} \quad (\mathrm{m}^2) \qquad (3)$$

where Q = volume flow rate of oxygen (m^3 s^{-1}), p and p_a = inlet and atmospheric pressure (absolute) (N m^{-2}).

Any one of the three relationships may be used depending on circumstances.

4. APPARATUS

The following specification of the apparatus is intended to give a precision of 1% in the determination of the specific permeability coefficient $K_{(\mathrm{oxygen})}$. This specificaton was found convenient and adequate for normal concrete but can be relaxed or tightened up depending on circumstances.

1. The general layout of the apparatus is shown diagrammatically in Fig. 1. The essential elements are: oxygen supply cylinder fitted with a pressure reducing valve, precision pressure regulator, pressure gauge, the permeability cell, flow meter and a stop-watch.
2. Commercially available oxygen supply cylinders fitted with a normal pressure reducing valve were found satisfactory.
3. The pressure regulator should be capable of controlling the inlet pressure to the cell over a range of absolute pressure from 1×10^5 to 6×10^5 N m^{-2} (1 to 6 bar) in stages of 0.5×10^5 N m^{-2} and to maintain a set pressure level within 1% of the selected pressure for at least 30 min.
4. The pressure gauge should indicate pressure with an accuracy of at least 0.6% of the total range of about 5×10^5 N m^{-2}. The graduations on the pressure gauge should be 5×10^2 N m^{-2} or less.
5. The cell should be designed to take operating pressures of up to 5×10^5 N m^{-2}. Three different cell designs are shown in Fig. 2. The essential features are as follows:

(a) a tightly fitting collar pressing against the curved surface of the concrete specimen to form a seal. The material from which the collar is made may be typically soft polyvinyl chloride (PVC), cold cast polyurethane rubber or some other;

(b) the clamp which tightly presses the collar against the specimen may be typically a rigid split ring held together by bolts, or a pneumatic pressure device such as a standard vehicle wheel inner tube located within a confined space and pumped up to a pressure of 5 to 15×10^5 N m^{-2}. The latter device can accommodate variations of up to 10 mm in the diameter of the specimen;

(c) the other essential parts of the cell are made of steel, aluminium or plastics and are clamped together with bolts;

(d) suitable small-bore pressure pipe and fittings

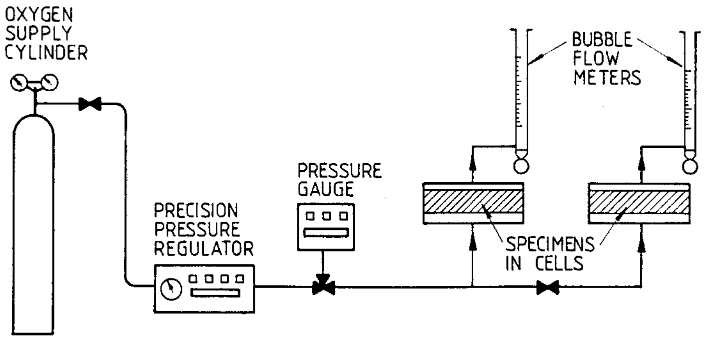

Fig. 1 Line diagram of the oxygen permeability apparatus.

appropriate for the maximum pressure employed should be used.

Cells complying with the above recommendations may be obtained commercially.*

6. The volumetric gas flow meter can be typically of the soap bubble type. The volume-calibrated glass tube should be of a cross-section such as to permit the measurement of a selected volume traversed by a bubble within a given time interval with a precision of not less than 1%. A number of bubble meters covering a range of volumes will normally be required to accommodate the range of concrete permeabilities under test. Typically, the volume of the flow meter will be in the range of 1 to 100 ml, with tube diameter in the range of 2 to 20 mm. The optimum bubble traverse time lies in the range of 20 to 60 s.

7. A stop-watch with a precision of time interval measurement of 0.1 s is adequate.

5. PREPARATION OF SPECIMENS

Cast or cored specimens may be used.

5.1 Cast specimens

The method may be used to characterize a concrete of selected materials, mix proportions, method of compaction, conditions of curing and subsequent exposure. In these circumstances the disc specimens are cast, ensuring always that they are homogeneous and truly representative of the selected parameters. The maximum aggregate size is restricted to 20 mm or 2/5 of the disc thickness.

The moulds for casting the discs should be of sturdy construction and made of rigid materials. Steel or rigid u-PVC have been found satisfactory. The size of the mould should be preferably 150 mm in diameter and 50 mm in depth. The dimensional tolerances should be preferably limited to 1 mm as this can be easily accommodated in the provisions of the specification of the permeability cell without the need for adjustment. Joints should be sealed to prevent leakage of mixing water or grout. Stiff petroleum jelly-type grease has been found suitable. Release agents, if required, should be used sparingly as their adherence to the specimen can influence the flow of oxygen and therefore the result.

Curing should be carefully laid down and strict compliance with the laid-down details should be ensured as the curing conditions of any concrete have a major influence on the permeability. If more than one disc specimen representing a given set of parameters is cast, care is required to ensure strict uniformity of treatment if a wide variation of results is to be avoided. It is recommended that a minimum of three specimens are used to obtain the permeability of cast concrete.

A standard reference method for the preparation of the disc specimens is as follows:

(i) compact the concrete on a vibrating table in a single lift, strike off the excess of concrete with a straight edge and cover with a plastic sheet;

(ii) cure for 24 h in a fog room at RH $>$ 95% and 20 ± 2°C;

(iii) demould the disc and wrap a closely adhering rubber sleeve around the cylindrical surface to prevent evaporation through this surface during subsequent drying (Section 5.3);

(iv) wrap the specimen in a plastic sheet, seal and store vertically (i.e. supported on the cylindrical surface) for 28 days at a temperature of 20 ± 2°C;

(v) remove the plastic and subject the specimen to the pre-conditioning regime as given below under Section 5.3.

5.2 Cored specimens

The method may be used to test concrete from a structure as represented by a cored specimen.

When selecting cores for the purposes of this test, care is needed to ensure that they have not been cracked or

*Details of the recommended cells as shown in Fig. 2 can be obtained from the following sources: Pneumatic seal Type (a): British Cement Association, Wexham Springs, Slough SL3 6PL, UK. Mechanical clamp Type (b) and pneumatic seal Type (c): Forschungsinstitut der Zementindustrie eV, Tannenstr 2, D 4000 Düsseldorf 30, West Germany. Type (c) was developed by Centre d'Études et de Recherches de l'Industrie des Liants Hydrauliques (CERILH), France. For any information, please contact M J. C. Duriez, Directeur des Recherches, Ecole Nationale Supérieure des Techniques Industrielles, 6, avenue de Clavières F-30107 Alès, France.

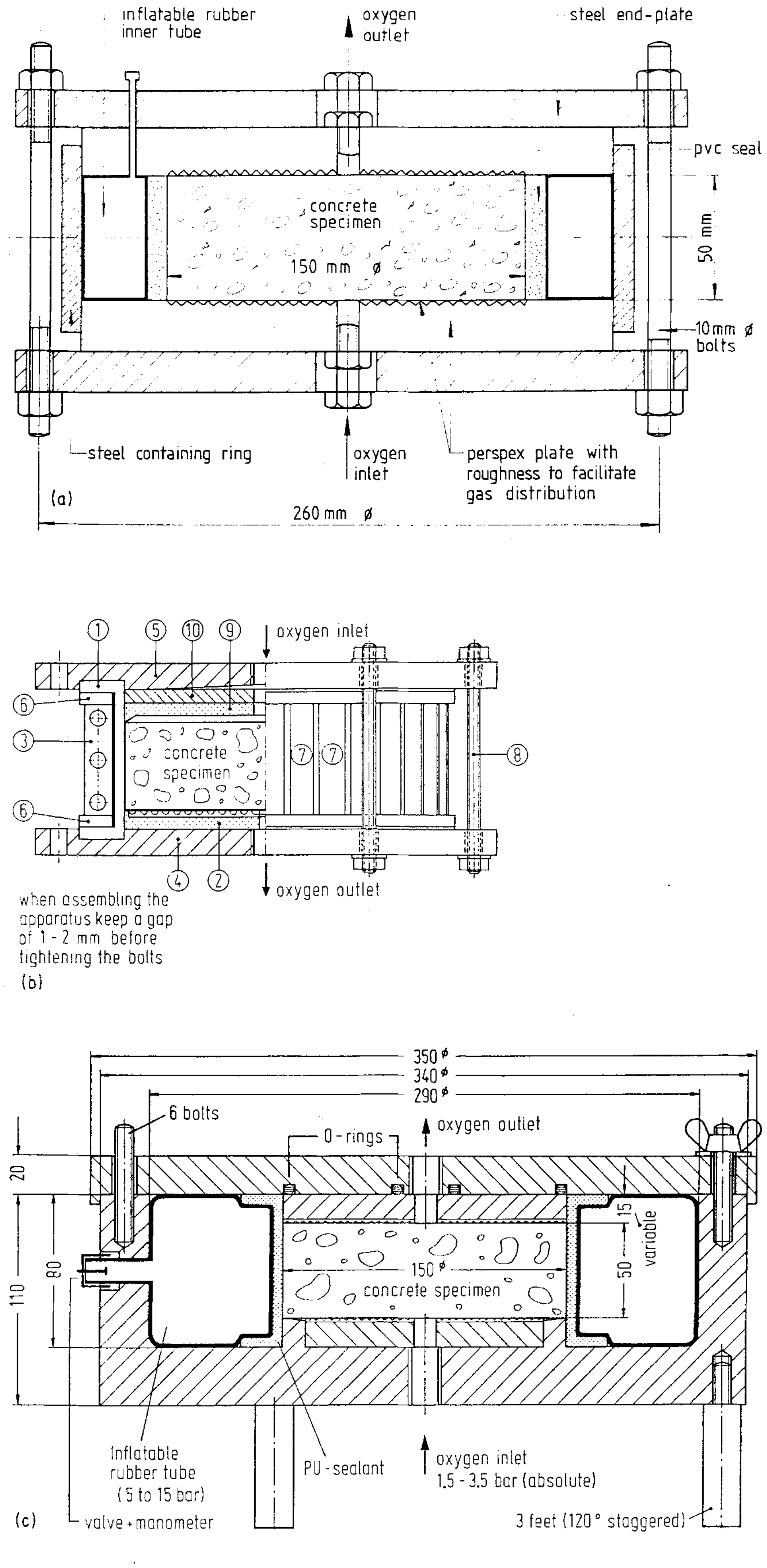

Fig. 2 (a–c) Details of the recommended cells in three versions, Types (a), (b) and (c). Key to Type (b): (1) sealant (PUR), Shore hardness scale A-55; (2, 9, 10) supporting plates, oxygen distributors (hard PVC); (3) clamp (split ring, 2 mm sheet steel, two halves closed by bolts); (4, 5) bottom and top plates (steel); (6) load distributors (hard PVC, four half rings); (7) supports (steel, 30 pieces); (8) bolts (steel, 6 pieces).

otherwise damaged in the cutting or slicing operations. The most convenient core diameter for this test is 150 mm, which can be cut into a disc specimen 50 mm thick.

Smaller-diameter cores and even irregularly shaped specimens can be used after an appropriate adjustment and embedment in a suitable impermeable resin to produce composite specimens with a reduced effective permeation area. Smaller thicknesses than the standard 50 mm can be accommodated in the permeability cell by the use of spacers. In the circumstances of reduced diameter and/or thickness coupled with the use of resin, the interpretation of the permeability results thus obtained will be burdened with uncertainties and should be more difficult. In any case the results obtained on far from standard specimens should not be directly compared with those obtained on standard cast or cored specimens without appropriate calibration.

5.3 Pre-conditioning

To obtain meaningful results the prepared specimens, whether cast or cored, must be brought to the same moisture condition before an oxygen permeability test can be carried out. Specimens saturated with moisture will not give a reading on the gas flow meter under the conditions of pressure difference employed in this test. Completely dry specimens are equally impossible to achieve.

The standard initial moisture conditions can be achieved in two different ways as follows:

Regime A. Store in a laboratory atmosphere at 20 ± 2°C and RH 65 ± 5% for a period of 28 days. Specimens should be supported on the cylindrical surface, ensuring a free movement of air around them.

Regime B. Dry in a ventilated laboratory cabinet at 105 ± 5°C for a period of 7 days, followed by storage in a desiccator for 3 days at a temperature of 20 ± 2°C.

Regime A is to be preferred as it simulates normally encountered exposure. Regime B also leads to a substantially higher permeability.

It should be understood that a deviation from the above-mentioned pre-conditioning regimes is acceptable provided all specimens in a given investigation are pre-conditioned in the same way, and provided only a relative performance of those specimens is required.

6. TEST PROCEDURE

The permeability test should be carried out in a laboratory controlled at 20 ± 2°C, RH 65 ± 5% and free from draughts.

Measure the thickness and diameter of the specimen disc with a precision of 0.5 mm. The mean of five measurements of each dimension is considered adequate.

Weigh the specimen. Although the mass of the specimen does not enter the determination of permeability, it is considered useful to know the mass in cases where the specimen is subject to a change in moisture condition over a long period of time and is subject to handling by operators.

Take the reading of the atmospheric pressure (p_a) with a precision of better than 5×10^2 N m^{-2} (5 mbar). Place the specimen in the cell and assemble the apparatus. Ensure that there are no leaks, particularly between the cylindrical surface of the specimen and the snugly fitting collar and in all fittings up to the bubble flow meter. Ensure that there is no obstruction to the pipe delivering the oxygen and all items of apparatus are fully operational.

Select five absolute inlet pressure stages in succession as follows: 1.5, 2.0, 2.5, 3.0 and 3.5×10^5 N m^{-2} absolute, and determine the exact pressure at each stage with a precision of better than 5×10^2 N m^{-2}. At each pressure stage allow the flow rate to stabilize, which is normally achieved within 5 to 30 min. Take provisional readings of the flow rate every 5 min. Repeat until the difference between successive readings is less than 3%. At that point take at least two readings in quick succession and record the flow (Q_i) for that pressure stage. Increase the pressure to the next selected stage and repeat the flow rate stabilization procedure. Repeat the determination of the flow (Q_i) at each pressure stage on the way down the pressure range. Evaluate the mean of the two flow rates ($\bar{Q}$). Calculate the coefficient K_i by substitution in Equations 1, 2 or 3. Evaluate the mean K from the five K_i values obtained for the five pressure stages.

It is recommended to plot Q_i against $(p^2 - p_a^2)$. This helps to demonstrate the extent of deviation of the results from linearity and helps to detect any outlying result due to, say, abnormal behaviour of the apparatus. A systematic deviation from linearity with the points lying on a smooth convex curve is normal. The convexity increases with the mean permeability of concrete. The deviation from linearity of the curve is mainly due to the inaccuracy in the assumption that the flow of oxygen through concrete is laminar.

7. TEST REPORT

The test should include the following:

(a) date of test and age of concrete;
(b) specimen – cast or cored;
(c) description of concrete;
(d) factual or circumstantial evidence regarding the history of curing regime to which the concrete was subjected;
(e) conditions and duration of pre-conditioning;
(f) temperature and relative humidity in test laboratory;
(g) dimensions and mass of specimen; and
(h) give the five K_i values for each of the five pressure stages and the specific permeability coefficient $K_{(oxygen)}$ for the specimen.

8. CONSIDERATIONS OF PRECISION

This aspect of the method has not been exhaustively covered by the work of the Committee. However, the following partial statements relating to the precision of the method may be found a useful guide by the user:

(a) Results are considered consistent if the individual results of five tests performed by the same operator in the same laboratory on the same specimen do not vary by more than 2% from the mean.

(b) A coefficient of variation of 4% has been achieved on a single specimen tested in seven laboratories.

(c) Concrete specimens exchanged between laboratories usually agree to within 20%.

(d) The single-operator coefficient of variation from 10 specimens cast from the same concrete batch has been found to be sensibly constant at about 30% over a range of 13 very different concrete mixes.

(e) Specified concrete mixes prepared by a prescribed method from the same materials in eight different laboratories in various countries revealed a large variation in the measured oxygen permeability. This, however, is not considered to be a reflection on the lack of repeatability of the method itself, but points to the difficulties in arranging and accurately specifying comparative tests on concrete in different laboratories across linguistic and geographic obstacles. Also, the insistence on homogenization of all primary materials as well as strict adherence to the laid-down procedure can scarcely be overemphasized in the conduction of such tests.

9. ACKNOWLEDGEMENT

The eight European laboratories participating in this study were: Cementa AB (Sweden), Cement and Concrete Association (UK), Blue Circle Industries (UK), Aalborg Portland (Denmark), Forschungsinstitut der Zementindustrie Dusseldorf (West Germany), Italcementi (Italy), Centre d'Etudes et de Recherches de l'Industrie des Liants Hydrauliques (France) and Cementfabriek IJmuiden BV (Holland).

10. BIBLIOGRAPHY

1. Klinkenberg, L. J., 'The permeability of porous media to liquids and gases', in 'Drilling and Production Practice', (American Petroleum Institute, New York, 1941) pp. 200–214.
2. Žagar, L., 'Die Grundlagen zur Ermittlung der Gasdurchlässigkeit von feuerfesten Baustoffen', *Arch. Eisenhüttenwesen* **26** (1955) 777–782.
3. Deutsche Normen DIN 51058, 'Testing of ceramic raw and semi-finished materials; determination of specific gas permeability of refractory bricks' (1963).
4. Därr, G. M. and Ludwig, U., 'Determination of permeable porosity', *Mater. Struct.: Res. Test.* **6** (33) (1973) 185–190.
5. Lawrence, C. D., 'Durability of concrete: molecular transport processes and test methods', Technical Report 42.544 (Cement and Concrete Association, Slough, 1981).
6. Grube, H. and Lawrence, C. D., 'Permeability of Concrete to Oxygen', in Proceedings of RILEM seminar on Durability of Concrete Structures under Normal Outdoor Exposure, University of Hanover, March 1984 (Institut für Baustoffkunde und Materialprüfung) pp. 68–79.
7. Gräf, H. and Grube, H., 'The influence of curing on the gas permeability of concrete with different composition', *ibid* pp. 80–87.
8. Lawrence, C. D., 'Transport of oxygen through concrete', in Proceedings of British Ceramic Society meeting on Chemistry and Chemically Related Properties of Cement, London, September 1984 (British Ceramic Society) pp. 277–293.
9. Grube, H., 'Influence of concrete materials, mix design and construction techniques on permeability', in Proceedings of Conference on Permeability of Concrete and its Control, London, December 1985 (The Concrete Society) pp. 77–88.
10. Lawrence, C. D., 'Laboratory testing of cores', *ibid* pp. 70–76.
11. *Idem*, 'Measurement of permeability', in Proceedings of 8th International Congress on the Chemistry of Cement, Rio de Janeiro, September 1986, Theme 4.1, Volume V pp. 29–34.
12. Dutron, P., Ody, R. K., Ursella, P., Grube, H., Bager, D. H., Tran-Thanh-Phat, de Vree, R., Malmberg, B., Lawrence, C. D., 'Cembureau co-operative programme on permeability measurements for concrete', in Proceedings of RILEM 40th Anniversary – 1st International Congress: from 'Materials Science to Construction Materials Engineering', Paris, September 1987, Volume 1: Pore Structure and Material Properties – Association Francaise de Recherches et d'Essais sur les Materiaux et les Constructions. Edited by Maso, J. C. (Chapman and Hall, London, 1987) pp. 45–48.