CONSEC 2010 - 6th International Conference on Concrete under Severe Conditions, Environment and Loading, 7-9 June, Mérida (Mexico), CRC Press, p. 1123- 1130.

# Analysis of sorted powder samples for the assessment of deteriorated concrete

R. Felicetti
*Department of Structural Engineering - Politecnico di Milano, Milan, Italy*

ABSTRACT: In this paper a new device is presented, aimed at collecting the powder ensuing while drilling a concrete member with an ordinary hammer-drill. The result is a sorted sample of ground material which keeps trace of the original depth of extraction in the concrete cover. The powder is contained in a test tube that can be adapted to the specific analysis for which the sample is intended (colorimetric inspection, measurement of the carbonation depth, thermal analyses, etc). The main advantages of this technique are the reduction of the damage caused to the structure, the very little time needs (one carbonation test takes about one minute) and the lower instrumentation and labour costs. These benefits and the reliability of the results are weighed against the established test methods for the same properties.

## 1 INTRODUCTION

Most of the external agents having an effect on the durability of concrete structures (freeze and thaw, fire, carbonation, alkali-silica reaction, etc) lead to more or less pronounced variations of the material properties within the concrete cover. For this reason point-by-point inspections are generally needed in order to characterize the profiles of the investigated material properties.

A common approach to the assessment of such gradients is based on the extraction of cores, to be examined as they are (visual observation, pH and colour measurement, ultrasonic scan) or to be cut into slices for subsequent laboratory testing, each slice being representative of the material response at a definite depth (LCPC 2005). Though very accurate and reliable, this methodology is rather time demanding and it can hardly extended to a statistical evaluation of the variability among the different parts of a structure.

Once the acquisition of cored samples has been planned, further data on the cover condition can be gained by fitting the core drill with a set of sensors in order to monitor the resistance encountered during the drilling process. As an example, in the assessment of fire damage the feed rate of the cutting tool proved to be quite a sensitive indicator of the residual strength (Fig. 1a, Felicetti 2009b). The interesting feature of this method is the ability to continuously scan the material at increasing depth, even in presence of steep gradients in the mechanical response of the concrete cover (Fig. 1b).

In this perspective, a faster and less invasive alternative is the inspection of the resistance encountered while drilling a small diameter hole (Ø = 10 mm) with an instrumented battery hammer-drill (Felicetti 2006). Compared to core extraction, the main limitation is the lack of a material sample to be submitted to further analyses, though the remaining hole and the ground-concrete powder could be in principle the object of additional investigations. As an example, pictures of the inner surface of the drilled cavity can be taken with the aid of an endoscope fitted with a digital camera. The visual observation can take advantage of a procedure for digital image processing that allows to work out a front view of the unwrapped cylindrical surface of the hole (Felicetti 2009b).

Concerning the possible utilization of the drilling debris, it has to be remarked that several

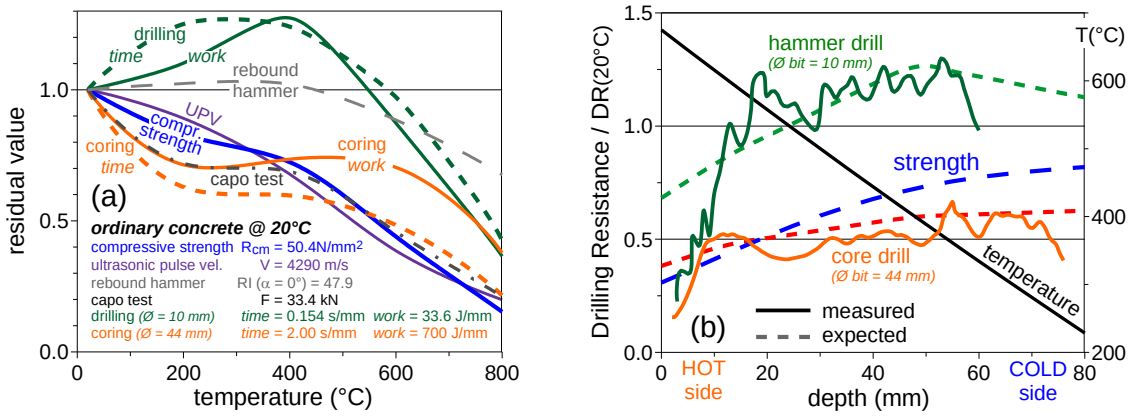


Figure 1. (a) sensitivity to a uniform thermal damage of some common NDTs and of the core- and hammer-drilling techniques and (b) profiles of the drilling resistance indicators in the case of a concrete panel exposed to a steep thermal gradient (675 to 230°C left to right - Felicetti 2009b).

types of analysis require (or allow) a preliminary grinding of the material into a fine powder. Hence, being able to take drilling-powder samples while keeping trace of their original depth in the concrete cover would be an efficient way to tackle the condition assessment problem. To this purpose, a common procedure consists in dividing the drilling process in regular steps (in the order of 10 mm) while carefully collecting the debris produced in each step. As a matter of fact this approach is rather time consuming and allows just a coarse resolution in the subsequent point-by-point analyses.

A promising alternative is represented by a dedicated device, which allows to continuously collect the powder streaming out from the hole mouth during the normal drilling operation. An account on its functioning and on the test that have been performed in order to check its accuracy and viability is given in the following sections.

## 2 A DEVICE FOR TAKING SORTED SAMPLES OF DRILLING-POWDER

Several physicochemical analyses on concrete require a preliminary grinding of the material into a fine powder (X-ray diffraction, chloride-ions content, Differential Thermal Analysis, Thermo-Gravimetric Analysis, etc - Fib 2008). Moreover, some tests that are normally performed on the intact samples, may be carried out also on the material in pulverized form (carbonation depth, colour measurement, etc). This evidence suggests to take advantage of the powder ensuing while drilling a concrete member with a common hammer-drill. In case of a monitored perforation, the concurrent measurement of the drilling resistance can be profitably merged with the following examination of the resulting powder.

Compared to the ordinary laboratory practice, the only limitation of this approach is the impracticality of controlling whether to include or not the coarse aggregate in the sample. In case of steep variations of the investigated properties with the drilling depth, an important requirement is to preserve the order of extraction, so to obtain a sorted sample of powder.

To this purpose a special device has been developed, which allows to gather the ground-concrete streaming through the helicoidal grooves of the drill bit. Basically, it consists of a annular head with a circular brush, to be pierced with the drilling tool (Fig. 2). The head is fitted with a funnel which converges the powder down into a vertical test tube. This latter can be adapted to the specific analysis for which the sample is intended. As an example, a transparent material allows to control the regular flow of the powder during drilling and to perform a first visual inspection. A narrow cut along the generatrix, thin enough to prevent the particles to pass, makes possible to infiltrate the sample with the liquid chemicals used in some material analyses (e.g. the phenolphtalein solution for the carbonation depth measurement).

Some preliminary tests were performed in order to check the factual sorting of the extracted powder. A number of layered specimens were prepared by gluing plates of different colours (bricks and stones - Fig. 3). These ones have been drilled horizontally and the ensuing powder has been collected in transparent methacrylate pipes. By analysing the digital images of these

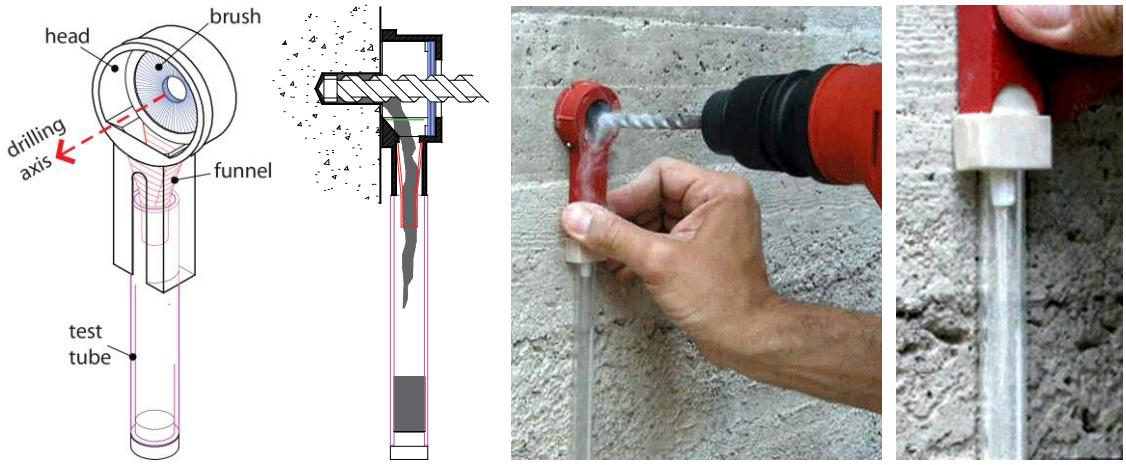


Figure 2. The device for taking sorted samples of drilling-powder: assonometric rear view and vertical section; the device in operation and detail of the powder flowing through the funnel (Felicetti 2009a).

samples in the RGB colorimetric space, it is possible to assess quantitatively the pureness of the powder based on its chromaticity.

The diagram in Figure 3 is an example of such representation concerning a calcarenite-brick specimen. For the sake of clarity the plots have been normalized in the range between 0 (calcarenite) and 1 (brick). It can be noted that the powder keeps a good pureness up to about 40 mm depth, with a sharp colour variation at the first layer change. Then an increasing influence of the lateral scratching of the hole is observed, leading to some mixing between the layers. However, this effect becomes sizeable in a range which is deeper than the cover thickness of most structures and it can be reduced by improving the alignment of the drill bit.

One last aspect to be considered while analyzing these samples is the scale factor between their length and the real depth of the drilled holes. Taking into account the lower apparent density of ground concrete compared to the pristine material, it has been found that a $Ø_{int}$ = 9 mm test tube combined with a 10 mm drill bit yields a scale factor of about 2, improving the sensitivity of the deterioration depth measurements. However, the precise determination of this ratio should be repeated after each sampling.

This device has been recently patented and reengineered, by optimizing the geometry of the different parts and by developing some new details aimed at improving its effectiveness in on-site applications (Felicetti 2009a). It is foreseen to make it available in a complete kit for the evaluation of the carbonation depth in concrete structures.

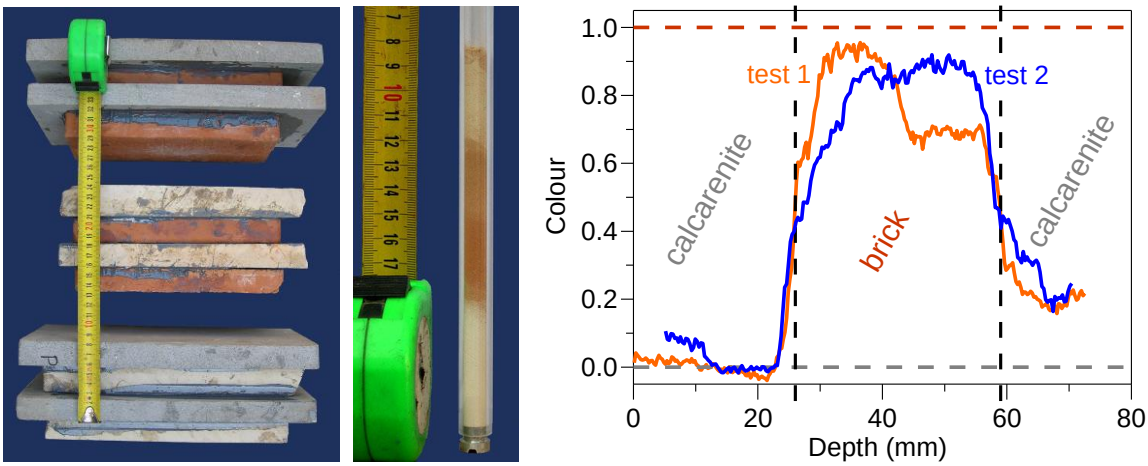


Figure 3. Layered specimens of alternate colours; powder sample extracted from the central calcarenite-brick specimen and diagrams of the colour variations along the powder sample (the abscissa is scaled to the actual depth in the drilled hole).

## 3 EXAMPLES OF DRILLING-POWDER ANALYSES

### 3.1 *Measurement of the carbonation depth*

In order to check the viability of the testing technique at issue, different types of analysis have been performed on the sorted samples of drilling powder (bit diameter = 10 mm). The first example concerns the measurement of the neutralization depth in a set of concrete cubes (side = 100 mm) submitted to an accelerated carbonation process. Some through micro-cores have been extracted from the cubes (Ø = 17 mm) and a pH indicator has been applied on their surface, highlighting the carbonated zones at the extremities (Fig. 4a). The same reagent has been infiltrated in the drilling powder samples via the thin longitudinal cut of the dedicated transparent test tubes. In this latter case the thickness of the carbonated zone has to be corrected by the ratio between the hole depth and the sample length (about 2 in this test series).

The results obtained with the two methods (7 and 10 mm at the two ends of the core vs. 6 and 8 mm based on the powder samples in Fig. 4a) are in reasonably good agreement, if one considers the local irregularities of the carbonation front due to the material heterogeneity (Fig. 4b). This latter effect is smoothed down in the powder sample, making easier the interpretation of the test result. Other remarkable advantages of the new method are the quickness (about one minute per test), the small operational requirements (no need for water and AC supply) and the lower instrumentation and labour costs compared to the conventional drilling of micro-cores.

It is then feasible to increase the number of the measurement points and to weigh the variability of this kind of deterioration in the case of real structures. One example is provided by the investigation carried out on the unprotected facades of a concrete church (St. Giovanni Bono in Milan, Italy - Fig. 5). This building dates back to the Sixties and already exhibited durability problems ascribable to the reinforcement corrosion, as testified by a number of existent repair patches. Based on the onsite test results (100 cover thickness measurements and 14 tests on powder samples) it is confirmed that the average depth of carbonation already exceeded the average cover thickness at the time of testing. The former parameter was also characterized a by significantly higher dispersion.

By assuming these variables are governed by independent Gaussian distributions, then also their difference is Gaussian and the probability can be assessed of having passed at any point the initiation period for steel corrosion (namely carbonation depth > concrete cover - Fig. 5b). In the present case a 75% probability is worked out, which is consistent with the diffuse signs of rebar corrosion of this building. An identical result can been obtained, with no assumptions on the probability distribution, by dividing the depth $x$ in 5 mm classes and applying the procedure indicated by Pentti & Mattila (COST 2003):

$$P_{init} = \Sigma_i \, P_{init} \, [x_i < x \leq x_{i+1}] = \Sigma_i \, \{ \, P_{cover} \, [x_i < x \leq x_{i+1}] \cdot P_{carbonation} \, [x \geq (x_i + x_{i+1}) / 2] \, \} \qquad (1)$$

where the probability P of either the cover or the carbonation depth to fall in a specific range is determined on the basis of the observed frequencies.

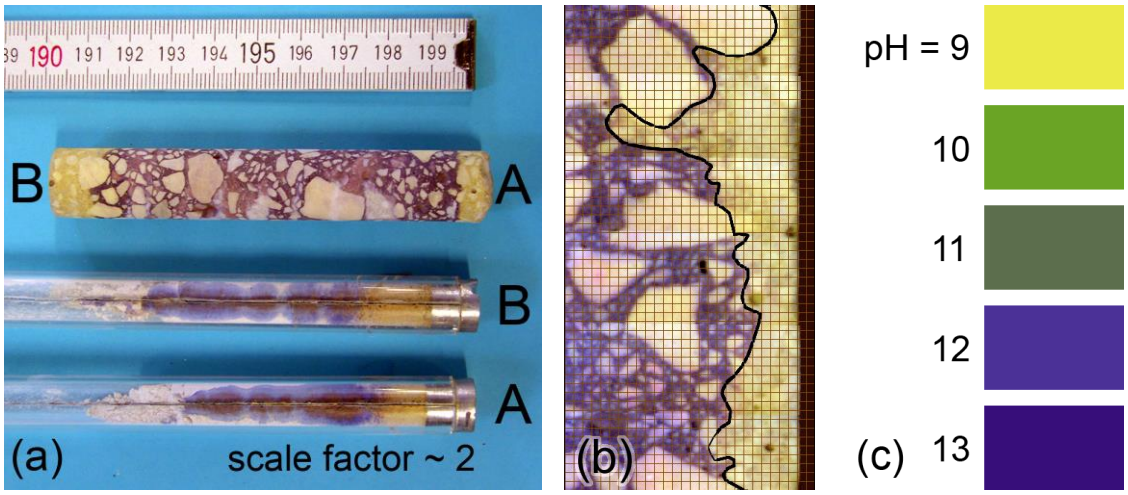


Figure 4 (a) Carbonation depth in a 100 mm concrete cube determined on a micro-core and on two sorted powder samples; (b) local irregularities of the carbonation front and (c) colour scale of the pH indicator.

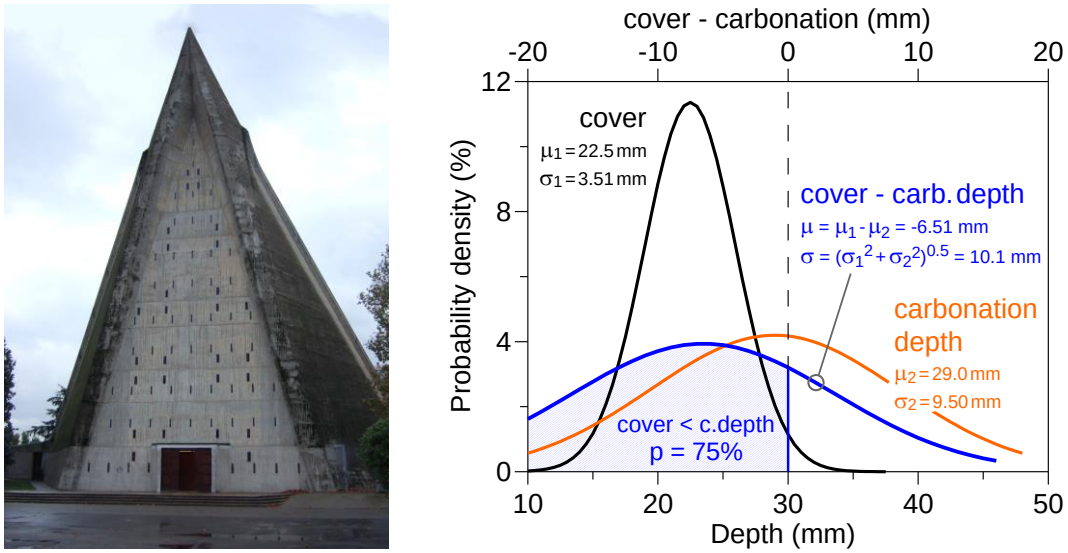


Figure 5. The St. Giovanni Bono church in Milan and comparison among the frequency distributions of the concrete cover, the carbonation depth and their difference.

### 3.2 *Colorimetric analysis*

Besides the distinct colour variation due to the pH indicators, the adoption of transparent test tubes allows also detecting the slight chromatic alterations experienced by deteriorated concrete. One important example is the discoloration induced by the exposure to high temperature, which is a recognized parameter for the assessment of fire damage. In this case the colour is expected to change at increasing temperature, generally from normal to pink or red (300-600°C), whitish grey (600-900°C) and buff (900-1000°C). The pink-red discoloration ensues from the possible presence of iron compounds in the fine or coarse aggregate, which dehydrate or oxidise in the indicated temperature range (Short et al. 2001). The strength of this colour change depends on the aggregate type and it is more pronounced for siliceous aggregates and less so for calcareous and igneous aggregates. Detecting this first colour alteration is of great interest because its appearance usually coincides with the onset of a significant loss of concrete strength as a result of heating (LCPC 2005).

In a former research (Felicetti 2005) a simplified approach to colorimetry has been formulated, based on the analysis of the side picture of a concrete core, taken via a low-cost digital camera. The starting point of this method is that digital pictures are usually not very accurate by the colorimetric point of view, but they still allow to recognize slight colour variations among different points on the same sample. In most of the analyses on deterioration processes the deepest part of the sample is formed by the pristine material and it can be taken for reference in the representation of the other points measurements.

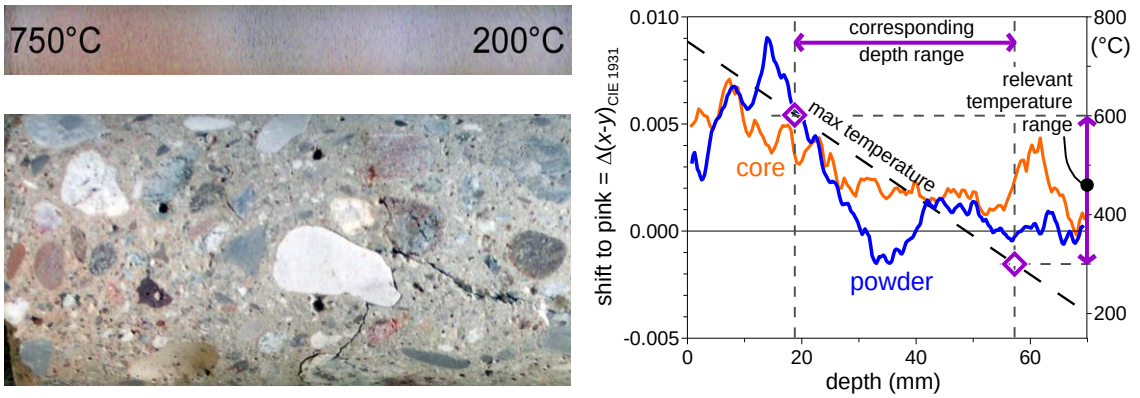


Figure 6 - Discoloration of both a powder sample and a core taken from the same heated panel and corresponding colour variation profiles obtained via the digital image analysis.

The same technique can be applied to digital images of the sorted powder samples, allowing to plot the profile of the colour shift towards pink at increasing depth within the heated member. The viability of this method has been checked with reference to a concrete panel exposed to a steep thermal gradient (about 8°C/mm - Fig. 6). Compared to the conventional analysis of cores, an increased noise is observed in the case of the powder sample, probably because of the random influence of the coarse aggregate. Nonetheless, the main features of the material alteration can still be detected in the relevant range from 300 to 600°C. Averaging the colour profiles resulting from different drilled holes would considerably improve the readability of the discoloration onset.

### 3.3 *Multiple Thermal Differential Analysis*

The last example regards the differential thermal analysis (DTA), that involves heating a small sample of powdered concrete together with a similar amount of inert material (e.g. aluminium oxide $Al_2O_3$). Both samples are monitored in order to trace their temperature difference, which ensues from the transformations occurring in the tested material. This method has been proposed as a way for analyzing fire damaged concrete, because during this second heating minor or different transformations occur until the maximum temperature already experienced during the fire is exceeded (Handoo et al. 2002). In the case a temperature profile through the cover has to be worked out, the DT analysis has to be repeated on a series of samples taken at increasing depth, which is quite a demanding procedure.

In order to overcome this limitation, a sorted sample of drilling-powder has been collected in a metal pipe by means of the special device above described. A small amount of aluminium oxide is then added on top of the extracted powder. The pipe (Fig. 7a) is made of a thermally stable alloy (nickel-chromium) and it is perforated at regular steps along one generatrix so to allow to vent the developing gases and to embed a series of thin shielded thermocouples in the inner powder (Fig. 7b). By heating the pipe in a split-tube furnace (5°C/min) several DT analyses can be performed in one take.

Though less rigorous than adopting the standard test procedure and a dedicated device, this method is far less time demanding and still allows to detect the onset of the relevant transformations. The first results (Fig. 7c), pertaining to a concrete wall exposed to fire on just one side, seem in good agreement with the trends reported in the literature (Alarcon-Ruiz et al. 2005). The anticipated dissociation, compared to unheated portlandite, of the calcium-hydroxide resulting from the calcium-oxide rehydration (350-500°C) and the disappearance of the peak ascribable to the calcium-carbonate dissociation (700-800°C) are the main features to trace on these thermo-differential plots.

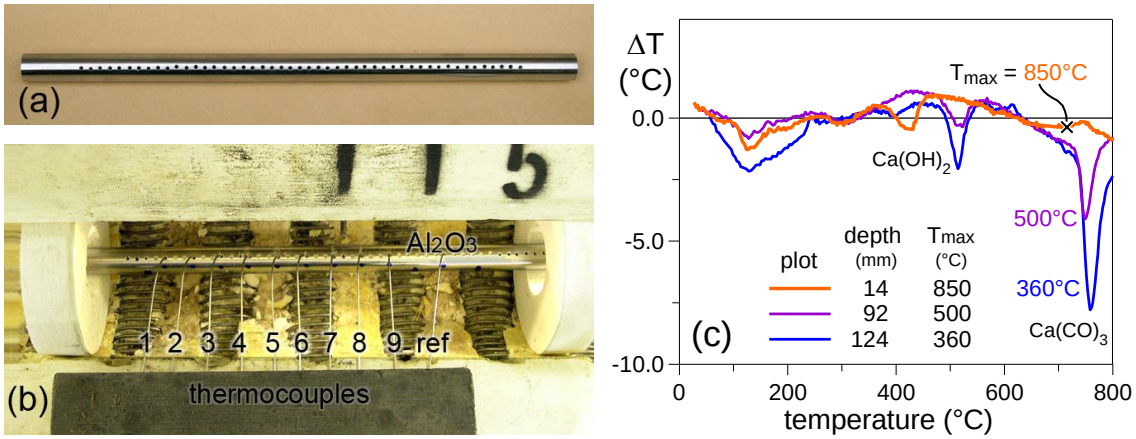


Figure 7. (a) Nickel-chromium perforated pipe to be filled with a sorted sample of drilling-powder and (b) test setup in the split-tube furnace; (c) temperature differentials pertaining to different depths in a heated panel.

CONSEC 2010 - 6th International Conference on Concrete under Severe Conditions, Environment and Loading, 7-9 June, Mérida (Mexico), CRC Press, p. 1123- 1130.

## 4 CONCLUSIONS

In this paper the idea to analyse the powder produced while drilling a concrete member is regarded as an alternative method to inspect the chemo-physical deterioration of the cover due to the environmental agents. To this purpose, a special device has been developed so to allow to continuously gather the powder streaming out from the hole mouth. Several tests have been performed in order to check its viability and possible applications. The main conclusions that may be drawn are summarized in the following.

The ability of the device to keep separated the drilling-powder extracted at each depth within the concrete cover is reasonably good in the range of interest for most applications. Some care is needed for relatively deep holes (> 50mm) so to limit the lateral scratching due to the possible misalignment of the drill bit.

Collecting the powder samples in transparent test tubes allows to control the sampling operation and to perform a first visual inspection on the material. With the aid of digital image analysis techniques, a deeper colorimetric inspection can be also carried out without any further preparation of the sample.

A customized test tube, including a thin longitudinal cut, makes possible the application of a pH indicator to reveal the carbonation depth. This is probably the most promising application of the proposed method. Due to the remarkably fast and easy implementation of the test, the variability ascribable to the local exposure conditions can be tackled in a consistent way.

More sophisticated tests may be also performed via specifically suited test tubes. One example is a set of Differential Thermal Analyses that are run in parallel in a thermally stable metal pipe. Moisture and ion penetration measurements are other common tests still to be addressed.

In summary, the main advantages of the proposed technique compared to the traditional drilling of cores are the minor impact on the structure, the small time and operational requirements and the lower instrumentation and labour costs. Conversely, it is not possible to control the effect of the coarse aggregate, whose local influence may sizeably affect the results. Averaging some repeated measurements is a possible solution to this problem.

According to the present trends in the Non Destructive evaluation of concrete structures, one interesting perspective of the analyses on the drilling-powder is their possible combination with other inspection techniques. Among them, the monitoring of the resistance encountered while drilling the concrete cover is the most natural integration of the method herein presented.

## REFERENCES

Alarcon-Ruiz L., Platret G., Massieu E. & Ehrlacher A. 2005. The use of thermal analysis in assessing the effect of temperature on a cement paste. *Cement and Concrete Research*, 35, 609–613.

Felicetti R. 2005. Digital-Camera Colorimetry for the Assessment of Fire-Damaged Concrete. In P.G. Gambarova, R. Felicetti, A. Meda & P. Riva (eds.), *Fire Design of Concrete Structures: What now? What next?; Proc. Int. Workshop fib TG 4.3*, Milan, 2-3 Dec. 2004, Brescia: Starrylink, 211-220.

Felicetti R. 2006. The drilling resistance test for the assessment of fire damaged concrete. *Journal of Cement and Concrete Composites*, 28, 321-329.

Felicetti R. 2009a. Improved procedure for the analysis of construction materials and device to implement this procedure. Patent Application MI2009A 001073, 1706/2009 *(www.tecnoindagini.it)*.

Felicetti R. 2009b. Combined while-drilling techniques for the assessment of the deteriorated concrete cover, In O. Abraham & X. Derobert (eds.), *7th International Symposium on Nondestructive Testing in Civil Engineering - NDT-CE'09*, Nantes (France), 30 June-3 July, 369-376.

FIB Bullettin n.46 2008. *Fire design of concrete structures - Structural behaviour and assessment*. In P.G. Gambarova & R. Felicetti, Chapter 8: *"Expertise and assessment of structures after fire"*, 64-114.

Handoo S.K., Agarwal S. & Agarwal S.K. 2002. Physicochemical, mineralogical, and morphological characteristics of concrete exposed to elevated temperatures. *Cem. and Conc. Res.* 32, 1009–1018.

LCPC Laboratoire Central des Ponts et Chaussées 2005. Présentation des techniques de diagnostic de l'etat d'un béton soumis à un incendie. Report ME 62, 114 p. (in French).

COST 521 2003, Corrosion of steel in reinforced concrete structures. In Cigna R., Andrade C., Nürnberger U., Polder R., Weydert R. & Seitz E. (eds), *Final Report - Publication EUR 20599*, European Communities, Luxembourg.

Short N.R., Purkiss J.A. & Guise S.E. 2001. Assessment of Fire-Damaged Concrete Using Colour Image Analysis. *Construction and Building Materials*, 15, 9-15.