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**Lawrence, M. and Walker, P. (2008) The Measurement of the Carbonation Profile in Lime Mortars using a Drilling Resistance Measurement System. In: Proceedings of the 11th International Congress on Deterioration and Conservation of Stone, 2008-09-15 - 2008-09-20, Toruń.**

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# THE MEASUREMENT OF THE CARBONATION PROFILE IN LIME MORTARS USING A DRILLING RESISTANCE MEASUREMENT SYSTEM.

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## Abstract

Air lime mortars gain strength through the development of carbonation over extended periods. This paper presents a method of measuring this strength development using a drilling resistance measurement system (DRMS)

Variations in drilling resistance as measured by DRMS were shown to map closely onto the carbonation profile as mapped using thermogravimetric analysis (TGA). The DRMS drilling resistance was shown to vary proportionately to the extent of carbonation across this front as measured by TGA to a high degree of conformity.

The DRMS technique has been shown to accurately measure the position and slope of the carbonation front, as well as providing a means of measuring the extent of strength gains achieved during carbonation. This technique can be applied in-situ causing only minor repairable damage to mortar.

**Keywords:** drilling resistance measurement system, DRMS, lime mortar, carbonation profile.

## 1. Introduction

The predominant binder in mortars, renders and plasters for use in the conservation of historic buildings is lime, in the form of either air lime or hydraulic lime. These binders gain strength over an extended period through carbonation where the portlandite in the lime changes to calcite, which in the case of some air lime mortars can take months or years. It is helpful to monitor this strength gain in order to understand the contribution that the mortar makes to the structural integrity of the construction. Phenolphthalein staining is the most commonly used technique to measure the extent of carbonation. This requires a freshly broken section of mortar, and the technique is not therefore easily applied in-situ. Other techniques such as Thermogravimetric Analysis (TGA) and Fourier-Transform Infrared spectroscopy (F-TIR) involve investigation using laboratory equipment. The use of a drilling resistance measurement system (DRMS) is investigated in this paper as a means of rapidly measuring in-situ the development of carbonation of lime mortars and of quantifying the extent of strength gain resulting from such carbonation.

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## 2. Theory

The strength gains over time seen in air lime mortars are the result of carbonation. Consequently the exterior of the mortar gains strength before the interior. The compressive strength development is expected to follow the carbonation profile. To verify this it is necessary both to map the carbonation profile using a chemical technique and to measure the compressive strength profile.

Compressive strength is generally measured by a direct uniaxial compressive strength test. This test measures the bulk compressive strength of a cube of material, and only accounts for variations in the strength at different depths through the cube in so far as those affect the bulk compressive strength. This test is unable to reveal any data about variations in the compressive strength within the matrix of the specimen.

A technique is available to measure changes in compressive strength through the depth of a specimen using the drilling resistance measurement system (DRMS) [Rodrigues et al, 2002]. This is designed to measure the force required to drill a hole at constant rotation (rpm) and lateral feed rate (mm/min). The force is known to correlate with the compressive strength of the material. When the rotation, lateral feed rate and type of drill bit are held constant, the measured force is an indication of the compressive strength of the material. Since the compressive strength of mortar varies according to the extent of carbonation, this system can be used to map changes in compressive strength and, as a result, changes in carbonation across the carbonation front. A standard test exists for cement mortar, which can be adapted for lime mortar [RILEM, 2004]. This system has been used to try to measure the effectiveness of stone consolidants [Lotzmann & Sasse, 1999]. Since the technique measures compressive strength, the study was not very successful, because the consolidants improved the tensile strength of the stone without changing the compressive strength. Where variations in compressive strength were being measured in fire damaged concrete [Felicetti, 2006], and in the decay of stonework [Rodrigues et al, 2002], the technique has proved to be reliable.

## 3. Experimental

The DRMS equipment used is produced by SINT Technology from Calenzano, Florence, Italy. This machine is one of the original 'Hardrock Project' machines [Tiano & Viggiano, 2000; Tiano, 2000; Fratini et al, 2006] which were used to evaluate the system by a number of establishments in several European countries.

The standard set-up for testing building stone uses a 5mm diameter purpose made diamond tipped drill with a flat tip. The rotational speed used is normally 600rpm and the rate of penetration 5mm min<sup>-1</sup>.

Trials were conducted using the standard set up and the results were found to be highly variable. The probable reasons for this were that mortar is a two phase material, consisting of binder with a compressive strength of between 0.5 N/mm<sup>2</sup> and 3.5 N/mm<sup>2</sup>, and aggregate with compressive strengths of between 20 N/mm<sup>2</sup> (oolitic limestone) and 60 N/mm<sup>2</sup> (silicate sand). In addition, the aggregate particles ranged from dust to 2mm in diameter, which was 40% of the diameter of the drill bit.

A number of trials were conducted using masonry drill bits varying in diameter from 7mm to 12mm, using rotational speeds varying from 300rpm to 1200rpm, and penetration speed varying from 3mm min<sup>-1</sup> to 15mm min<sup>-1</sup>. The most consistent results were found to be produced by a 10mm diameter masonry drill bit at 900rpm with a

penetration speed of  $5\text{ mm min}^{-1}$ . All data presented in this paper are gathered using this optimized set up.

### 3.1 Materials and methods

The experiment was designed to compare the results from DRMS with the carbonation profile as measured by TGA following the technique developed by Lawrence et al (2006). Tests were conducted on a specimens made using crushed bioclastic limestone and 4 month-old lime putty with a binder:aggregate (B:Ag) ratio of 1:3 prepared according to BSEN 1015-11:1999. Testing took place at 14, 28, 90, 180 and 360 days from the date of manufacture.

Six DRMS tests were conducted on each specimen and TGA tests were conducted at 3mm intervals through the depth of the specimen to establish the chemical carbonation profile. The TGA data on  $\text{Ca(OH)}_2$  content were converted into percentage carbonation figures and presented graphically against depth from the surface of the specimen. These data, at each time interval were superimposed on the DRMS data for comparison.

### 3.2 Data Reduction

The procedure for producing a DRMS curve of drilling resistance vs. distance from the surface involves several stages. The first stage is the gathering of the primary data. This is gathered at a resolution of 0.1 mm. Fig.1 shows all six data sets on the same graph for the 90 day old lime mortar.

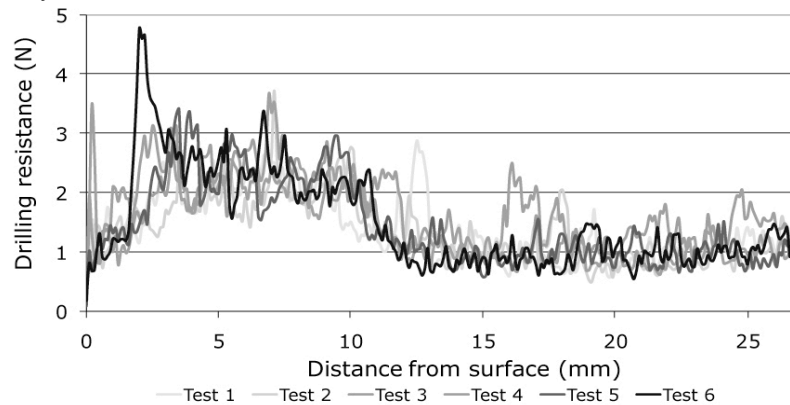


Figure 1: Raw DRMS data for 90 day-old lime mortar

It can be seen that the signal is relatively noisy. Occasional data points can be up to 100% different from the trend, but the majority of the data points are within 25% of the trend. It can be seen that there are common patterns in all data sets.

Firstly there is steady increase in drilling resistance over the first 2-3 mm. This is not due to a weaker outer layer, but rather to the shape of the drill bit. The tip is conical in shape and over the first 2.8 mm the drilling resistance will vary according to the depth of penetration since the area of drill in contact with the specimen varies from a point initially to  $\sim 90\text{ mm}^2$  when the cone has fully penetrated. Secondly there appears to be a plateau between  $\sim 3\text{ mm}$  to  $\sim 10\text{ mm}$ , followed by a steady reduction to  $\sim 13\text{ mm}$ . Finally there is a further plateau to the end of the test. This change is caused by a change in

drilling resistance, and the shape of the curve is that which would be expected from the carbonation front.

The noise that can be seen comes from two different sources. The material under test, lime mortar, is a two phase material. It is made up of binder, with a compressive strength of between  $\sim 0.5 \text{ N/mm}^2$  and  $\sim 3 \text{ N/mm}^2$ , and aggregate with a compressive strength of between  $\sim 20 \text{ N/mm}^2$  and  $\sim 60 \text{ N/mm}^2$ . As the drill penetrates through the matrix, it will encounter different proportions of binder and aggregate, depending on the particle size of aggregate present. This will result in different localised drilling resistance, and therefore produce 'noise' on top of the average drilling resistance of the matrix at any particular point. The other source of variations in drilling resistance are any voids present in the matrix. When the drill bit encounters these voids, drilling resistance will be reduced.

A certain amount of this noise is cleared up by the software which records the data as supplied by the manufacturer. The data reduction involves a smoothing process using averaging of a number of points. The average is calculated using a mobile window consisting of a number of points entered by the operator. Each point in the curve is replaced by the value obtained from averaging the points which precede it and those which follow it in a number established by the operator.

The final stage in the DRMS system data reduction process is to make an average of all six readings. This is done using the manufacturer's software by means of a simple average of the data from all six readings at each distance increment. This average can be seen represented by the dotted line in fig. 2.

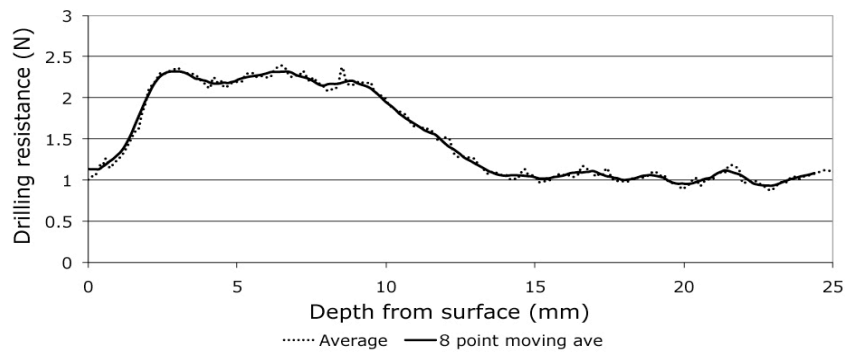


Figure 2: DRMS data - Averaged by the system (dotted); 8 point moving average to remove noise (solid).

This dotted line represents the final data reduction as recommended by the manufacturer. As can be seen, there is still a certain amount of noise present, and a further data reduction has been applied by the author in Excel®. The data reduction involves an 8 point moving average centred around each point. The aggregate is taken of the four force measurements before the point in question and the four force measurements after the point in question, and the mean is calculated. The same process is reiterated for each point. These data are shown in the solid line in fig.2. The shape of the curve is the same as the original curve, but the majority of the noise has been removed. This curve still shows the variation in drilling resistance over the initial

2.8mm caused by the conical shape of the drill tip. The rationale for this final data reduction is that it allows the data to be interpreted more easily whilst still closely following the line of the manufacturer's data reduction.

#### 4 Results and discussion

It is simplistic to assume that the reading at the location at which the drill tip fully penetrates the material represents the drilling resistance over the first 2.8 mm. The position of the drill, as recorded by the DRMS machine is the position of the point of the drill bit. The drilling resistance force recorded at this location is necessarily lower than the drilling resistance recorded when the full width of the drill has reached the same point. A first approximation of the location of the drilling resistance measurement might be 2.8 mm behind the point of the drill, when the full diameter of the bit has reached this location. This ignores the resistance imposed on the conical point of the drill bit in front of this location. The most rational location to take would be the point at which the same surface area of the conical bit is beyond the position as in front of it. This would occur when 45 mm<sup>2</sup> of the drill bit is in advance of the location and 45 mm<sup>2</sup> is behind it. This occurs 1.96 mm behind the point of the drill bit. Fig.3 shows these three data sets plotted graphically against the TGA data.

It can be seen that a 1.96 mm shift of the DRMS data maps closely onto the TGA data for the initial plateau, and the slope of the carbonation front. The second, lower plateau seen in DRMS does not map onto the TGA data. This is probably because there is insufficient calcite present to affect the drilling resistance of the mainly portlandite binder in the matrix.

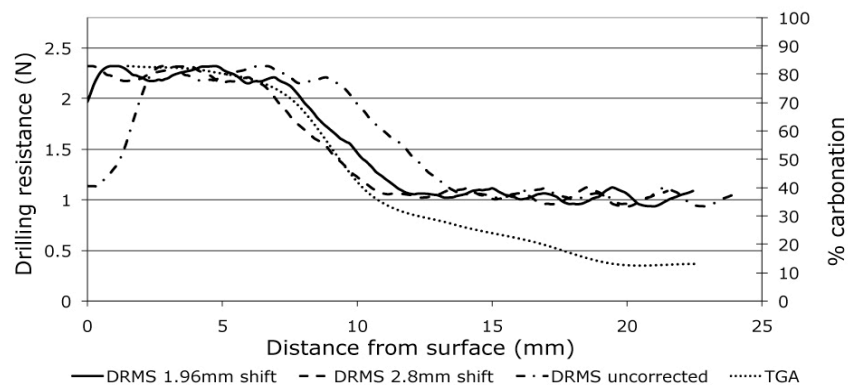


Figure 3: TGA carbonation data superimposed on adjusted DRMS data at 90 days.

All DRMS data used hereafter have been treated as described above using the additional data reduction technique and shifted by 1.96 mm backwards along the x-axis. Fig.4 to fig.8 show the TGA data presented graphically and superimposed on DRMS data as adjusted by the process outlined above for all 5 time periods. Fig.4 shows that at day 14 a small amount of carbonation can be seen in the TGA data over the first 5 mm. The DRMS data shows higher drilling resistance over the first 3 mm. The underlying drilling resistance seems to be unaffected by carbonation levels below ~40%. By day 28

(fig.5) the carbonation front as measured both by TGA and DRMS has progressed to ~5 mm. As with the 14 day data, the underlying drilling resistance seems to be unaffected by carbonation levels below ~40%. By day 90 (fig.6), the carbonation front is well developed, and the DRMS curve follows the TGA carbonation curve closely until the carbonation percentage goes below ~40%.

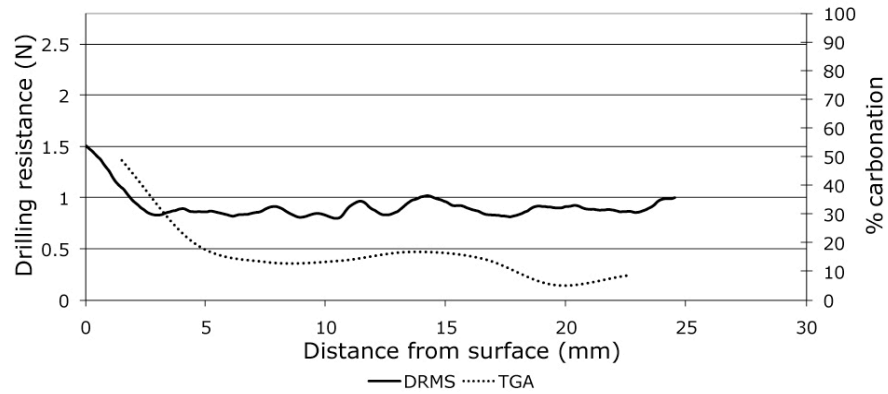


Figure 4: TGA carbonation data superimposed on adjusted DRMS data at 14 days.

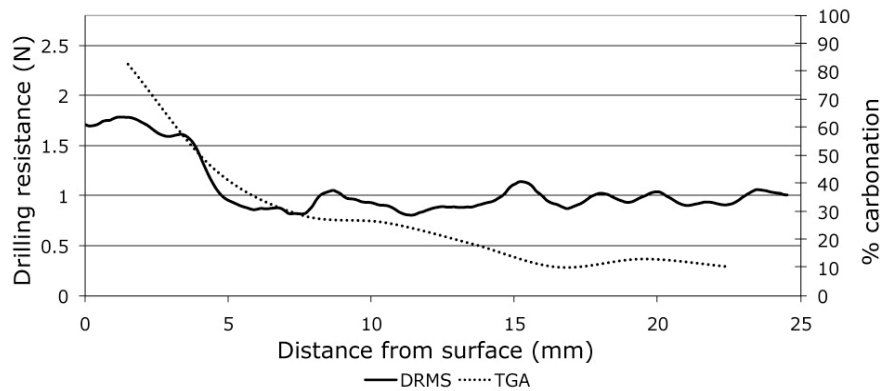


Figure 5: TGA carbonation data superimposed on adjusted DRMS data at 28 days.

The 180 day DRMS data (fig.7) once again follows the TGA data closely, and once again deviates once the carbonation percentage reduces to below ~40%. By day 360 (fig.8) it could be considered that the mortar is fully carbonated and there is little variation to be seen either in TGA data or in DRMS data. It should be noted that the TGA data shows some residual uncarbonated material. This is attributed to crystals of portlandite that have been enclosed by an impervious shell of calcite crystals making them inaccessible to atmospheric CO<sub>2</sub> as described by van Balen (2005).

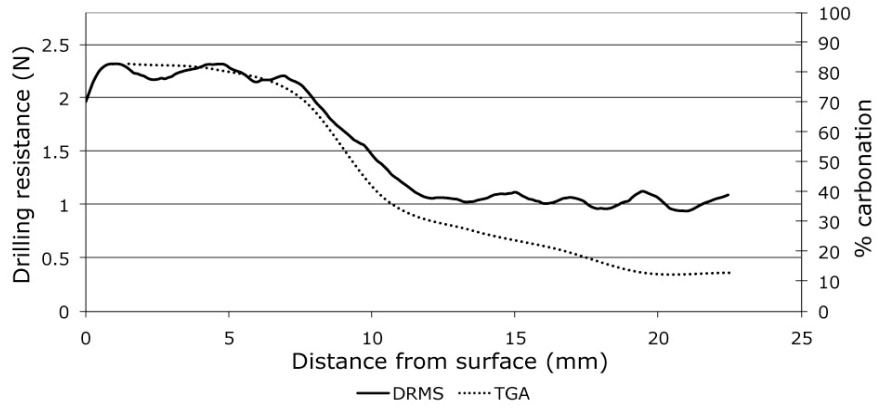


Figure 6: TGA carbonation data superimposed on adjusted DRMS data at 90 days.

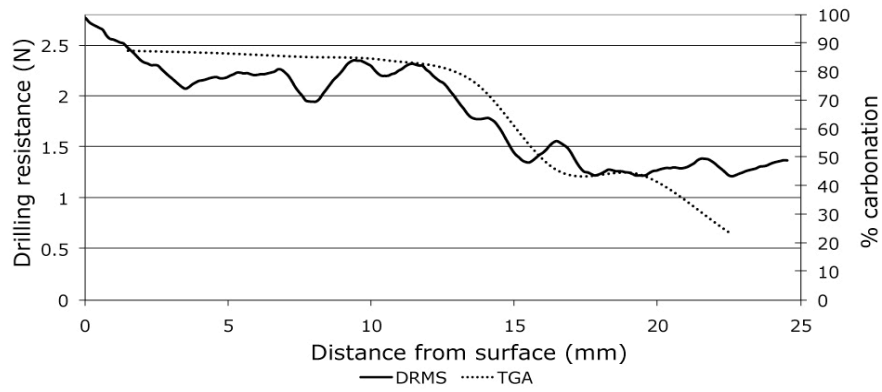


Figure 7: TGA carbonation data superimposed on adjusted DRMS data at 180 days.

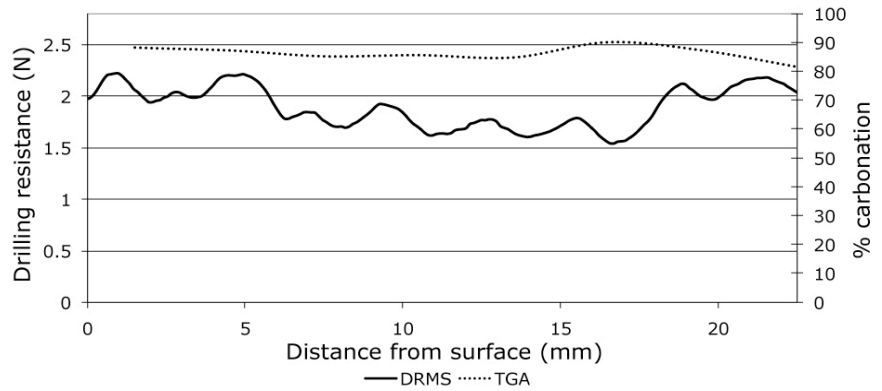


Figure 8: TGA carbonation data superimposed on final adjusted DRMS data at 360 days.



## 5 Conclusions

The experiments outlined above demonstrate that DRMS is capable of detecting and measuring the carbonation front. It would seem that when more than 60% of the portlandite remains uncarbonated in the matrix (at a 1:3 B:Ag ratio), the drilling resistance of the matrix is unaffected by the calcite that has been formed through partial carbonation. Above this level, DRMS closely follows the shape of the carbonation front. This is a useful characteristic, since DRMS testing can be performed in the field over a short period of time without recourse to laboratory testing such as is required for TGA.

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