

## Advanced Instrumentation for Civil Engineering Applications

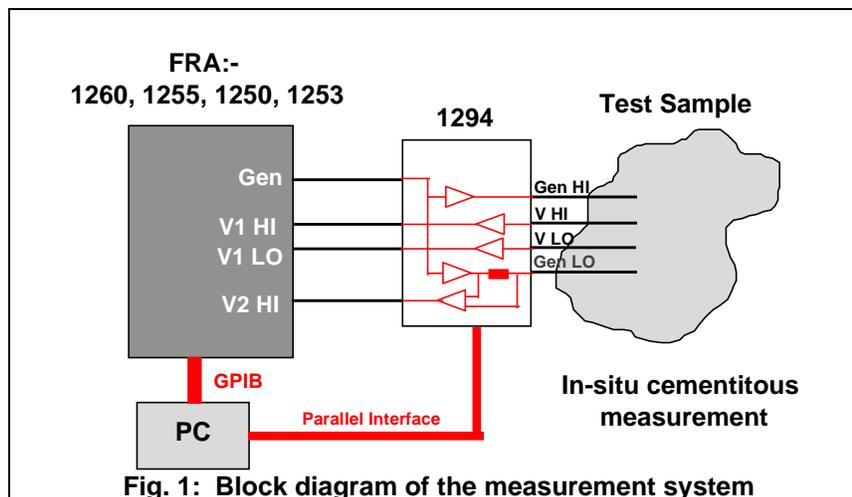
A. J. Hinton and B. Sayers:  
Solartron, Victoria Rd., Farnborough, UK

Electrical characterisation techniques have been adopted by the civil engineering community to investigate and monitor the development of cement microstructure. This has enabled the quality control of concrete at all stages of construction which is necessary to determine its durability.

Investigative studies have shown the compositional dependence of cement-aggregate-water systems with respect to their complex impedance. Researchers have been able to draw conclusions about the types of cementitious binders used, the bulk impedance has also been linked to the aggregate content of a system. The majority of measurements that have formed the basis for this research were taken between 1Hz - 10MHz.

Numerous traditional techniques have been used to study cement microstructure; these include scanning and transmission electron microscopy (SEM, TEM), nuclear magnetic resonance (NMR), small angle X-ray scattering and mercury intrusion porosimetry (MIP). Many of these do not lend themselves well to the direct investigation of the capillary pore network in cements, primarily due to sample preparation techniques, sample size and test conditions. Impedance Spectroscopy has advantages over these more traditional techniques including,

- non-destructive measurement
- non-invasive technique
- no special sample preparation required, impedance measurements can be made on larger samples therefore it is easier to examine the bulk properties of the material
- standard temperatures and pressures (r.t.p) are used, therefore cement microstructure is not disrupted, this is particularly important in the early stages of hydration



Impedance techniques have been used for numerous years in civil engineering to monitor corrosion in steel reinforced concrete (rebar corrosion). This has now been extended to the actual characterisation of cement microstructure, much of the work to date has centred on the later stages of hydration (after setting and during the hardening process). The ratios of individual components; such as ordinary Portland Cement (OPC) and pulverised fuel ash (PFA) and their effect on these processes has also been studied in the early stages of hydration. Electrical characterisation techniques in civil engineering are providing vital information about microstructure and performance, it is envisaged that their use will continue to grow.

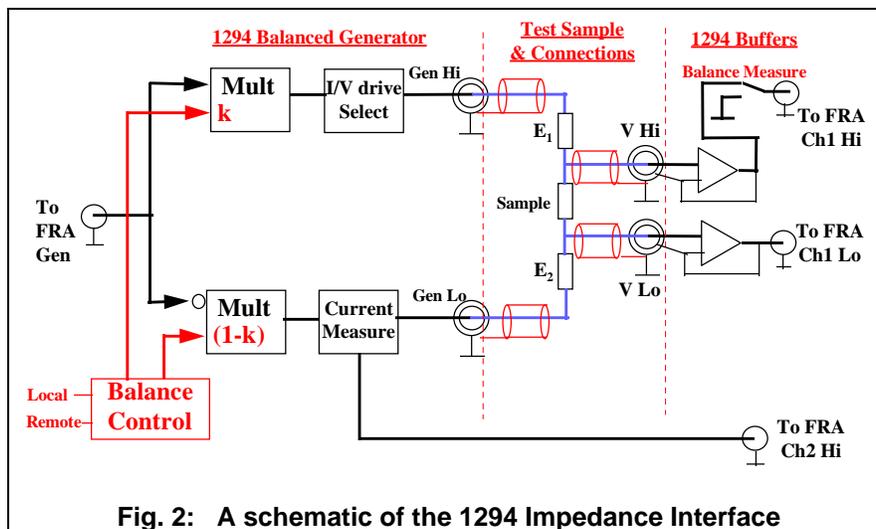
## Measurement Techniques

Previous research has centred on the use of the Solartron 1260 FRA for data collection. It was noted that there is a need to improve this technology specifically for four terminal measurement applications such as the measurement of cementitious systems. Four terminal measurements allow more accurate measurement of the impedance of the concrete mixture by rejecting impedance contributions from the electrodes. This led to the development of the 1294 which extends the analysis capabilities of the Solartron 1260. The specification of the 1294 Impedance Interface is achieved by the use of various techniques including 4-terminal connections to the sample, balanced generator and driven shield voltage measurement connections. In addition, the 1294 makes use of a sensitive multi-range current to voltage converter which allows the measurement of very low current levels which are commonly experienced in high impedance analysis or when using micro-electrodes.

The Solartron 1294 operates in conjunction with a frequency response analyzer (FRA), which provides the AC stimulus signal and correlation analysis of the output signals from the 1294. The use of correlation analysis is very important for the rejection of harmonics which result from non-linearities in the samples being measured.

The combination of instruments and associated software provides:-

- Impedance measurement range to >100 Gohm (2-terminal mode), 100Mohm (4-terminal mode)
- Wide frequency range 10 $\mu$ Hz to >1MHz
- Increased measurement accuracy by the use of 4-terminal driven shield connections and balanced generator techniques
- Temperature control using external controller (e.g. LakeShore 340 / Oxford Instruments) and cryostat (e.g. Oxford Instruments)
- Flexible PC software allowing the control of complex experiments at a range of frequencies, stimulus levels and temperatures, and the ability to plot results in a wide variety of formats.



**Fig. 2: A schematic of the 1294 Impedance Interface**

The schematic (Fig. 2) shows some of the features which have been incorporated to provide accurate and reliable measurement of electrical impedance. The sample is positioned between V Hi and V Lo, either side are electrode impedances represented by E<sub>1</sub>, E<sub>2</sub>. These impedances are due to the sample connections which are present in many civil engineering applications. There are also other stray impedances due to connections which may introduce imbalance in the electrode impedances which need to be considered and compensated.

The main features included in the 1294 are:-

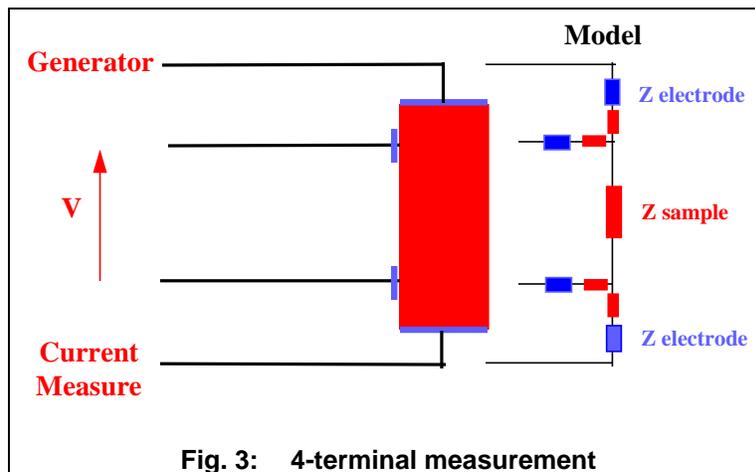
- low capacitance voltage measurement inputs using driven shields
- balanced generator for better common mode rejection
- built in attenuator for low voltage / current outputs
- accurate and sensitive current to voltage converter

### Measurement Techniques (4-terminal connections)

For civil engineering measurements, the electrodes which connect the instrumentation to the sample may be high impedance compared to the impedance of the sample itself. If conventional 2-terminal measurement techniques are used, there is no way to measure the impedance of the sample without including the impedance of the electrodes. This can lead to inaccuracies in the measurements, additional “features” on the impedance plots and to problems interpreting the data.

The four electrode connection technique (Fig. 3) uses separate electrodes for current stimulus and voltage measurement. This allows measurements of the sample on its own and minimises errors which would otherwise be introduced by the impedance of the current carrying electrodes. If 4-terminal techniques are employed, no current flows through the voltage measurement electrodes, which means that no voltage is dropped across these electrodes, leading to more accurate voltage measurements across the sample.

In addition, there may be localised disturbances where the current is injected into the sample (GenHi and GenLo). Four terminal techniques allow the voltage measurement electrodes to be placed well away from these disturbances in a region of linear electric field giving increased measurement accuracy.



### Driven shield connections to the sample

Accurate measurements at high frequency are a specific problem due to errors introduced by input and cable capacitance. One technique which has been widely used to reduce the effects of capacitance is to position electrometer buffer amplifiers close to the sample. These buffers are able to drive the cable and input capacitance of the equipment and therefore reduce errors in voltage measurements. However, these external amplifiers require a power supply which usually involves extra cabling. In addition, where the temperature of the sample is to be varied, the accuracy of measurements may be effected since the buffers must be positioned close to the sample and are therefore subject to the same temperature variations.

An alternative solution is the use of driven shield cables. This technique replicates the signal waveform (which appears on the cable inner), onto the cable shield in order to minimise leakage current flow between the cable inner and the shield. Since no current flows between the cable inner and shield, the impedance appears to be very large and therefore the effects of the cable and input capacitance are minimised.

This method allows the high impedance buffers to be kept within the instrument. As an external power supply unit is not required for the electrometers, the cabling is reduced and the sample temperature may be changed without affecting the accuracy of the results.

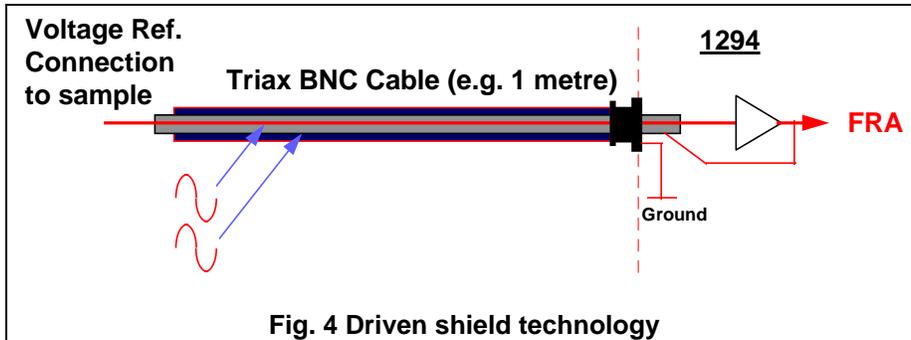


Fig. 4 Driven shield technology

### Balanced Generator

One problem associated with four terminal measurements on cementitious systems is the requirement to study the impedance of the sample in the presence of relatively high electrode impedances (i.e. to reject the voltages across the electrodes in order to obtain accurate measurements of the voltage across the sample itself).

Figure 5 shows a typical measurement situation where the electrode impedance is ten times higher than the sample impedance which is required to be measured. In the example shown, the sample impedance is simulated by a 1Kohm resistor, and the electrode impedances are simulated by two 10kohm resistors. Using conventional measurement techniques, the AC stimulus voltage is applied to GenHi while GenLo is grounded, and the current through the sample and voltage drop across the sample (between V Hi and V Lo) are measured in order to compute the impedance of the sample.

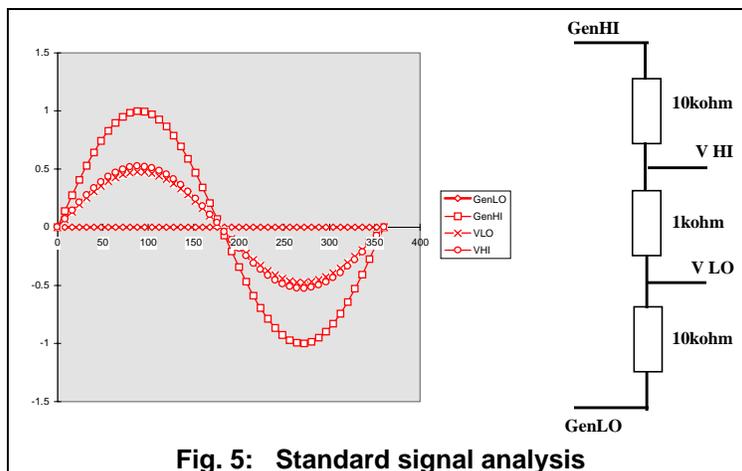
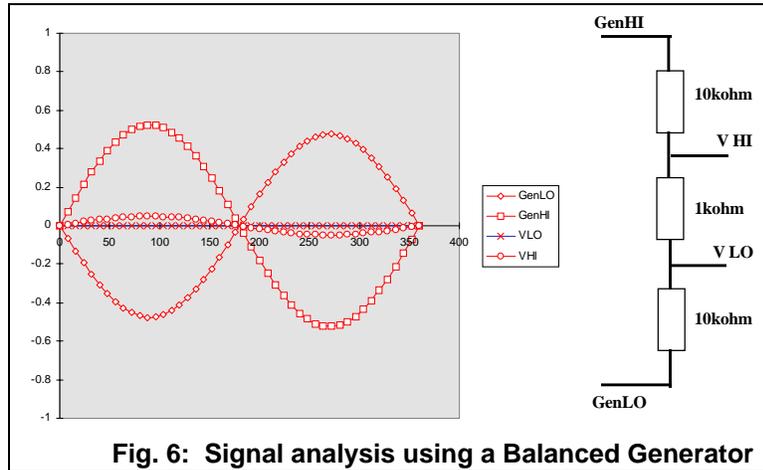


Fig. 5: Standard signal analysis

In this example, the FRA is required to measure a small voltage difference between two relatively high voltage signals which appear at V Hi and V Lo. For example, if 1 volt is applied between GenHi and GenLo, less than 50 mVolts appears across the sample (V Hi - V Lo) whereas the voltage on the V Lo measurement connection is approximately 500mV. The FRA is therefore required to measure a 50 mVolt "difference" signal in the presence of 500 mVolts which leads to errors which are referred to as "common mode" errors. In

addition, the relatively high voltage on the V Lo connection causes some current which has passed through the sample impedance to leak to earth via the input / cable capacitance on V Lo instead of being measured by the current measurement circuit.

The use of a balanced generator (see Fig. 6) reduces errors due to common mode voltages and earth leakage allowing more precise measurements of the sample impedance. This is achieved by adjusting the GenHi and GenLo signals in order to provide a balanced stimulus to the sample which has the effect of making the voltage which appears at V Lo as close as possible to earth voltage, (i.e. zero volts).



The balanced generator can also cope with extremely difficult measurement situations where for instance the electrode impedances are not equal. This is also typical for civil engineering measurements where it is difficult to obtain reproducible electrode contact. In this case the signals on GenHi and GenLo are set to different voltage levels in order to again achieve zero volts on the V Lo connection.

### Software capabilities

The software developed for use with this system is compatible with Windows 3.1 / 95/ NT4. It contains a variety of functions which allow maximum flexibility in terms of measurement and presentation of results, including:-

- Control of complex experiments involving multiple frequencies, stimulus levels and temperatures
- Presentation of results in a wide variety of formats including impedance, admittance, capacitance; with the ability to plot these as a function of frequency, stimulus level or temperature
- Multiple graph overlays allowing easy comparison of results taken at different frequencies or temperatures; or overlays of previously collected data
- Driver for Oxford Instruments and LakeShore Temperature controllers

### **The future of civil engineering measurements**

Research studies so far have concentrated on determining the compositional dependence of cement-aggregate systems in terms of their complex impedance. Replacement of OPC with PFA as shown unique changes in the nature of the impedance spectrum which can be directly correlated to the percentage levels of PFA present in the mix. It has also been shown that the bulk resistivity of a system is related to the fraction of cement paste present in the mix.

The technology incorporated into the Impedance Interface will push the boundaries of measurement capabilities for cementitious systems. The range of current applications provides an indication of the vital data that this instrumentation can provide in sample analysis. The trend in civil engineering research will continue to open up further areas which will benefit from electrical characterisation techniques to determine parameters such as; impedance, admittance, permittivity and phase/depression angle.

It is envisaged that electrical characterisation techniques will form the basis for quality control procedures to be adopted in the future by civil engineers. The quality control of cementitious materials is essential to ensure the requirements of function and durability are met in a construction industry which uses over one billion tonnes of concrete annually.

### **Further reading**

1. W. J. McCarter, The Application of Impedance Spectroscopy to Cementitious Systems, Solartron Technical Report No 29, May 1995
2. W. J. McCarter, Adv. Cement Res. The fractal surface of cementitious materials determined by impedance spectroscopy, **6**, 24, 147-154, 1994
3. W. J. McCarter and G. Starrs, Impedance characterisation of ordinary Portland cement-pulverised fly ash binders, J Mat. Sci. Let., **16**, 605 - 607, 1997
4. W. J. McCarter, A parametric Study of the Impedance Characteristics of Cement-Aggregate Systems during early hydration, Cement and Concrete Res., **24**, 1097-1110, 1994.
5. W. J. McCarter and G Starrs, Quality control of Structural Concrete: Assessing Fly-ash Content, Pro. Canmet/ACI Int. Conf., Kuala Lumpur, 1997

**For further details**

**Web:** <http://www.solartron.com/lap>

**E-mail:** [lab\\_info@solartron.com](mailto:lab_info@solartron.com)

**UK**

Victoria Rd., Farnborough  
Hampshire GU14 7PW England  
Telephone +44 (0) 1252 376666  
Fax +44 (0) 1252 544981

**USA**

964 Marcon Blvd. Suite 200  
Allentown, PA 18103, USA  
Telephone: +1 610-264-5034  
Fax +1 610-246-5329  
Toll-free 1-800 CALL SOL

**France**

37 rue du Saule Trapu  
91882 MASSY, Cedex, France  
Telephone +33 (0) 1 69 53 63 53  
Fax +33 (0) 1 60 13 37 06

**China**

Beijing Liaison Office  
Room 327, Ya Mao Building  
No. 16 Bei Tu Chen Xi Road  
Beijing 100101  
Peoples Republic of China  
Tel: +86 10-62381199 ext 2327  
Fax: +86 10-62384687