TechnicalNOTE

Verification of Low Impedance EIS Using a 1 mΩ Resistor

Introduction

Electrochemical Impedance Spectroscopy, EIS, is a very powerful way to gain information about electrochemical systems. It is often applied to new electrochemical devices used for energy conversion and storage (ECS), including batteries, fuel cells, and super-capacitors. EIS can be useful in all stages in the development of new devices, from initial evaluation of half-cell reaction mechanisms and kinetics, to quality control of packaged batteries.

Increased use of ECS devices in higher power applications (such as electric vehicles) has led to development of devices having very low impedance. Unfortunately for practitioners of EIS, impedance of modern ECS devices is often so low that it cannot easily or accurately measured using laboratory EIS systems. Most commercial EIS systems do not work well when impedance is below 0.1 Ω.

If an EIS system can make a measurement on a low impedance system, how can you tell if that measurement is valid?

This Technical Note describes a series of EIS measurements made on a 1mΩ surface mount resistor. A resistor with a very small shape was used to minimize cable related inductance errors. These measurements verify that a Gamry Instruments system can accurately measure this low impedance.

The techniques used to make the measurement and to correct for impedance errors due to the cabling will prove useful in verification of any EIS system used for low impedance measurements.

If you're new to EIS, you might want to read Gamry Instruments' **Basics of EIS** before reading the rest of this technical note. It can be found in the **App. Note** section on www.gamry.com. Information found in this introduction to EIS will not be repeated here.

Mutual Inductance

The cell-cable and placement of the leads connecting to the cell can have a major effect on EIS system performance. A phenomenon known as mutual inductance can limit the ability of an EIS system to make accurate measurements at low impedances and high frequencies. Mutual inductance errors appear in the measured EIS spectrum as an inductor in series with the cell's impedance.

This section describes mutual inductance and its effect on EIS measurements and offers practical suggestions for its minimization.

All high-performance EIS systems use a four-terminal connection scheme. The four leads that connect to the cell under test are grouped into two pairs.

- One pair of leads conducts the current between the cell and the system potentiostat. These leads will be called the current carrying leads.
- A second pair of leads measures the voltage across two points in the cell. These leads will be called the sense leads.

The term mutual inductance describes the influence of the magnetic field generated by the current carrying leads on the sense leads. In essence, the current carrying leads are the primary of a transformer and the sense leads are the secondary. The AC current in the primary creates a magnetic field that then couples to the secondary, where it creates an unwanted AC voltage.

This effect can be minimized in a number of ways:

- Avoid higher frequencies.
- Minimize the net magnetic field generated by the current carrying leads.
- Separate the current carrying pair from the sense pair.
- Minimize pickup of the magnetic field in the sense leads.

Avoid High Frequency
Mutual inductance creates a voltage error given by:

$$Vs = M \, di/dt$$

Vs is the induced voltage on the sense leads, M is the coupling constant (with units of Henries), and di/dt is the rate of change in the cell current.

M depends on the degree of coupling and can range from zero up to the value of the inductance in the current carrying leads. Assuming a constant amplitude waveform in the primary, di/dt is proportional to frequency.

The importance of the error voltage depends on its size relative to the true voltage being measured, which in turn is proportional to the cell impedance.

Mutual inductance errors appear in the measured EIS spectrum as an inductor of value M in series with the cell's impedance.

Minimize the Net Magnetic Field
A current passing through a wire creates a magnetic field with the field strength proportional to the current. Fortunately, passing the same current in opposite directions through adjacent wires tends to cancel the external field.

Two different wire arrangements are commonly used to minimize inductance and magnetic fields. The first is a coaxial cable; a central conductor is used to carry the current in one direction and a second conductor surrounding the first carries the current in the opposite direction. The second common arrangement is the twisted-pair; two insulated wires carrying current in opposite directions are twisted together.

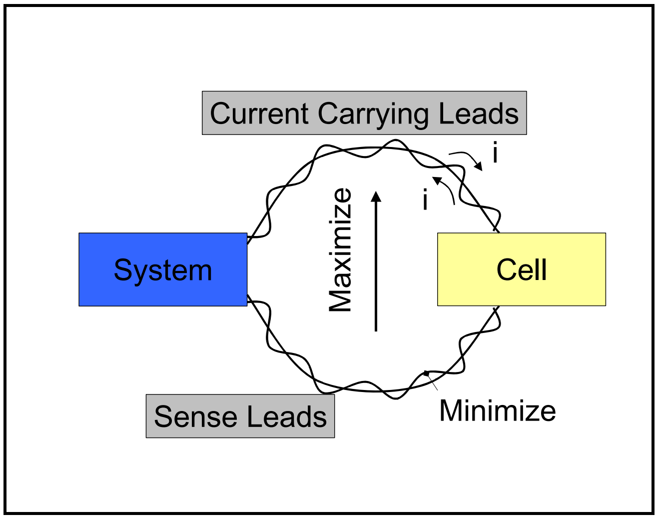
Separate the pairs
The magnetic field produced by a wire loses intensity as the inverse square of the distance away from the wire. Separating the sense wires from the current carrying wires can dramatically reduce the magnetic coupling.

Twist the Sense Wires
The concept of a magnetic loop probe is useful in understanding why a twisted sense pair minimizes magnetic pickup. A loop of wire in a changing magnetic field will see a loop voltage proportional to the area of the loop.

Twisting the sense wires helps in two ways. First, the twisted wires are forced to lie close to each other, minimizing the loop areas. Secondly, adjacent loops pick up opposite polarity voltages, which results in cancellation.

Cabling Recommendations
Use coaxial cable or twisted pair for each pair. The distance between the pairs should be maximized. Arrange each pair so that they approach the cell from opposite directions as shown in Figure 1.

Figure 1
Recommended Cell Connections



Mutual inductance errors are more significant at lower cell impedances and higher frequencies.

For example, on a system with 1 mΩ of resistance and 1 nH of mutual inductance, EIS phase shift will be 0.4° at 1 kHz and 3.6° at 10 kHz. If the resistance is lowered to 200 μΩ without changing the inductance, the phase shifts are 1.8° at 1 kHz and 17° at 10 kHz.

To minimize mutual inductance errors, Gamry Instruments has developed special twisted-pair cell cables for our EIS systems. The results below show how one of these cables improves the EIS spectrum of a test resistor.

Guidelines for Low Impedance EIS

These guidelines can greatly improve the accuracy of EIS measurements on low impedance cells:

- Use galvanostatic mode EIS.
- Use a large excitation current.
- Use twisted-pair or coax wiring.
- Use a low impedance cell surrogate to measure cable related impedance errors.
- Subtract the surrogate's spectrum from the cell's spectrum to correct for cable errors.

Each of these will be discussed below. Experimental data will illustrate the importance of these guidelines.

Experimental

The Resistor

The 1 mΩ resistor used in these measurements is part number WSL20101L00FEA18 from Vishay.

This resistor was designed for surface mount PCB applications. It is an industry standard 2010 size, with dimensions of approximately 5 mm long by 2.5 mm wide. Its accuracy rating is ± 5 % of its nominal value.

Vishay does not specify the inductance of this resistor. They do claim that the inductance of the WSL resistor family is between 0.5 nH and 5 nH and claim the family has "excellent frequency response to 50 MHz".

Electronics and Software

All experimental data were collected using a Gamry Instruments EIS300 EIS System built around a Reference 600 Potentiostat/Galvanostat/ZRA. In most of the tests, a Gamry Instruments Reference 600 Low Impedance Cell Cable, Gamry Part Number 985-81, was used in place of the standard cell cable.

The small size of the resistor used in these experiments makes direct connection of the resistor with the cell cable very difficult. Four 2.5 cm long pieces of 30 AWG Tefzel insulated wire-wrap wire were soldered to the ends of the Low Impedance cell cable. The solder joints were covered with polyolefin heat tubing and the small wires were twisted.

All tests were run using the Galvanostatic EIS script, with zero DC current and 350 mA of excitation current. The peak-to-peak current is approximately 1 Ampere. Unless otherwise noted, the EIS frequency sweep began at 0.1 Hz and ended at 1 MHz.

Shorted Lead Experiment

The shorted lead experiment was run with all four leads of a Low Impedance cell cable soldered together at one point. The cable had been modified by the addition of small diameter wires as described above.

The sense pair and current carrying pair were kept twisted as each pair approached the connection point from opposite directions.

Resistor Connections

Early attempts at these experiments yielded irreproducible results. It was very difficult to keep the connection lead geometry constant when the resistor was changed.

This was addressed by soldering the resistor across 2 pins of a 30 mm long header with 4 mm spacing between the leads. The 0.5 mm square pins of this header were held in place by two plastic spacers.

The bottom side of the resistor was soldered to the middle of the header pins. At least 10 mm of header pin stuck out on either side of the resistor.

Once the resistor was in place, the plastic spacers were slid off of the header pins and the last 4 mm of each pin was bent at right angles. The pins were then poked through holes in a piece of perforated fiberglass board, which held the assembly onto the board. Perforated fiberglass material is commonly used in electronics prototyping.

When a Low Impedance cable was connected to this assembly, the only 2 mm of each twisted pair was untwisted. The pairs approached the assembly from opposite sides of the resistor. Each wire was soldered to the appropriate gold plated pin about 5 mm away from the resistor.

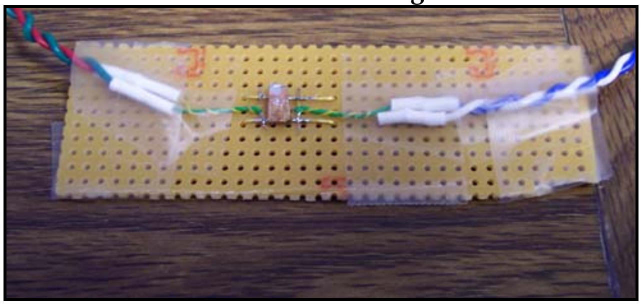
Once the wires were in place, they were scotch taped to the board. This prevents changes in the lead geometry when the assembly is handled.

Resistor Surrogate

A resistor surrogate was built to have the same geometry and to connect to the EIS system in the same way as the resistor. It consisted of a 5 mm by 2.5 mm slug cut from 1.6 mm thick copper sheet.

It was soldered to the same header used for the resistor measurements. Figure 2 is a photograph of the connections between the surrogate and the Low Impedance Cell Cable. The assembly looks crude, but it gave reproducible results.

Figure 2
Connections to Surrogate



Why Galvanostatic Mode?

Potentiostatic EIS is the most commonly used EIS technique. It is poorly suited to impedance measurements of ESC devices.

Current, voltage, and impedance are related through Ohm's Law. A voltage of 1 mV across 1 mΩ of impedance corresponds to 1 A of current.

No commercial potentiostat is specified to control a typical ESC device's potential (0.5 V to 4.2 V) with less than 1 mV of error. When a potential with a 1 mV (or larger) error is applied to a low impedance ESC device a very large DC current will flow.

Conversely, a galvanostat can easily control ampere currents to an accuracy of a few milliamps. The voltage on the cell is unaffected when the galvanostat is connected. A modern EIS system with AC coupling or offset and gain in the voltage measurement can measure 10's of microvolts of AC voltage superimposed on a stable device voltage.

For these reasons, Galvanostatic EIS is the preferred technique for EIS on ESC devices. This study employed Galvanostatic EIS to better model ESC measurements.

Why use Large Excitation Currents?

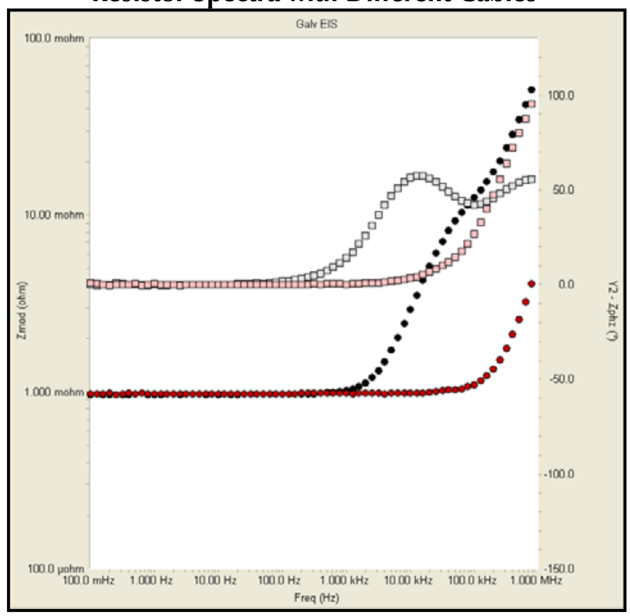
In a galvanostatic EIS experiment, the voltage signal is proportional to the applied current. Measurement of voltages smaller than ten µV is difficult, since most measurement systems have a few µV of noise.

It is best if the AC excitation current is kept large enough that the AC voltage is at least 10 µV. For a 1 mΩ cell, this means the current must be 10 mA or greater.

Why Use Twisted Pair Wiring?

Figure 3 shows the importance of wiring in EIS measurement of the 1 mΩ resistor. There are two Bode plots overlaid in this graph. In all Bode plots, the dark colors are magnitude and the corresponding light colors are phase.

Figure 3
Resistor Spectra with Different Cables



The black and grey data were recorded using the Reference 600's standard cell cable. Its alligator clips were attached to the pins on the resistor/header assembly. The red and pink data were recorded with a Low Impedance cable for the Reference 600 connected as described above.

Both curves have the same basic shape, and low frequency impedance very close to 1 mΩ. Both show inductive behavior at higher frequencies. The inductance is much lower with the Low Impedance cable.

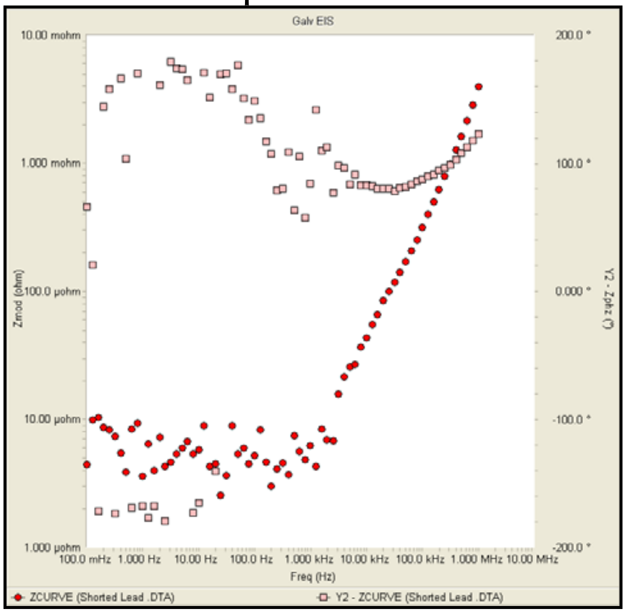
The Cable's Shorted Lead Spectrum

Figure 4 is the spectrum recorded on a Low Impedance Cable with all the leads soldered together.

This represents the lowest possible impedance measurable on this system. Notice that the low frequency phase is very noisy, but seems to tend toward –180° and +180°.

A good fit to a series RL model demands low frequency phase be close to 0°. This shorted lead spectrum therefore does not fit to an RL model. At higher frequency, the behavior is that of an imperfect inductor. Note that the phase at 1 MHz exceeds 90°.

Figure 4
Shorted Lead Spectrum with Low Z Cable



The Resistor Surrogate's Spectrum

Earlier graphs and discussion showed the importance of cabling on the measurement. But, even for the curve recorded using the low impedance cable, one doesn't know how much of the measured impedance is the true resistor impedance and how much to attribute to cable effects.

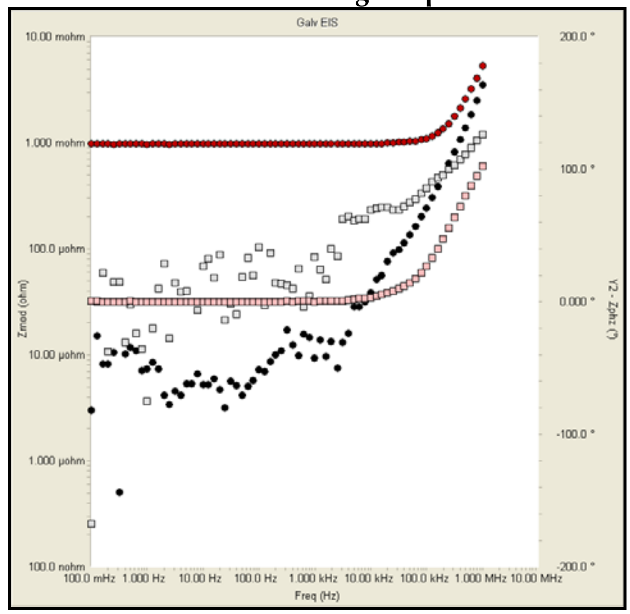
A resistor surrogate allows you to measure the cabling effects. The surrogate is a metal object with the same geometry and connection scheme as the resistor. It should be built to have as little resistance and inductance as possible.

The spectrum of a copper slug used as a surrogate was recorded using the same wiring and experimental conditions as the resistor test. Figure 5 shows Bode plots of the surrogate (in black and grey) and resistor spectra recorded using the same connection scheme and test conditions.

The surrogate spectrum is resistive at low frequencies and becomes inductive at higher frequencies. After removal of the point with an impedance below 1 µΩ, these data fit fairly well to a series RL model, with R = 4.5 µΩ and L = 444 pH.

Ideally, the surrogate's resistance should be zero. The estimated resistance of the copper slug using the bulk resistivity of copper and the slug's dimensions is 10.4 µΩ, (assuming path length of 2.5 mm). The thick layer of solder on the slug may partially explain the discrepancy between the 4.5µΩ experimental value and the 10.4 µΩ calculated value.

Figure 5
Resistor and Surrogate Spectra



Is Spectrum Subtraction Useful?

Resistive and inductive cabling errors caused by imperfect connections both result in impedance in series with the cell's true impedance. A series subtraction of the surrogate's spectrum from the resistor's spectrum can remove these effects.

Figure 5
Corrected and Uncorrected Battery Spectra

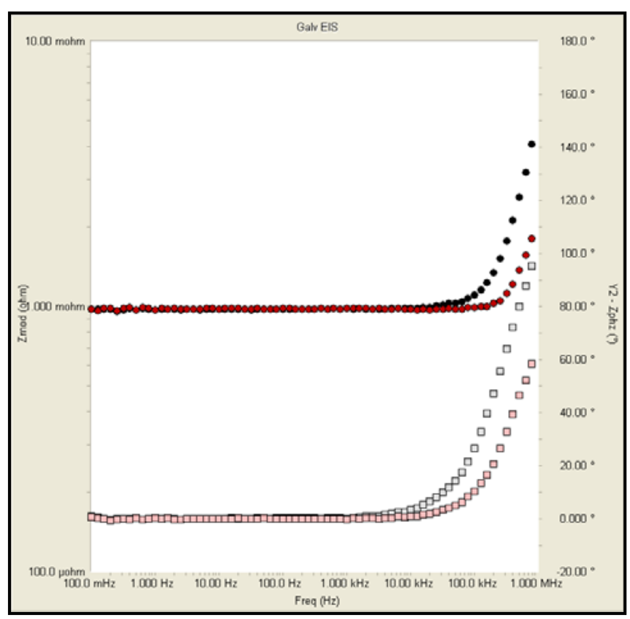


Figure 5 shows the resistor's spectrum before (in black and grey) and after (in red and pink) a series subtraction of the surrogate's spectrum.

The resistive region in the spectrum after correction obviously extends to higher frequencies.

Spectrum Comparisons

There were three resistor spectra discussed above.

1. The spectrum using the standard cell cable.
2. The spectrum using the Low Impedance cable.
3. The Low Impedance cable spectrum corrected using surrogate data.

All of these spectra were fit to a series RL model. In the case of the data recorded with the standard cell cable only the data below 10 kHz was used in the fit.

The R and L values resulting from the fits are shown in this table:

	R	error	L	error
Standard cable	978 μΩ	± 3.0 μΩ	53 nH	± 0.4 nH
Low Z	972 μΩ	± 2.6 μΩ	806 pH	± 65 pH
Low Z corrected	969 μΩ	± 2.6 μΩ	390 pH	± 3.8 pH

All the resistance values are within the 5% tolerance of the resistor.

Unlike lower value resistors, where the terminal resistance is negligible, the terminal resistance on this 1 mΩ part must be substantial. If the part were made from copper with the same dimensions, its end-to-end resistance would be about 50 μΩ, or 5% of the resistors value.

The resistance values from the fits may be low because a significant layer of solder built up on the resistor's terminals, which would lower the component's value. In addition, the resistor was specified assuming a distributed contact onto printed circuit board pads. Our connection geometry is quite different.

Notice that the resistance value calculated using the corrected spectrum is lower than the uncorrected data by about 3 μΩ. This is close to the 4.5 μΩ resistance value calculated from the surrogate's spectrum.

The method of subtracting a surrogate cell's impedance assumes that the surrogate has zero resistance. If it does not, the subtraction will cause an error, as seen here.

Gamry Instruments' Accuracy Contour Plot defines two accuracy regions. The first is the region that can be measured with less than 1% magnitude error and 2° phase error. The second is the region that can be measured with less than 10% magnitude error and 10° phase error.

What is the highest frequency that lies in each Accuracy Contour Map region on our three resistor spectra?

The limits are shown in this table:

	1% and 2°	10% and 10°
Standard cable	79 Hz	505 Hz
Low Z cable	5.0 kHz	31.6 kHz
Low Z corrected	12.6 kHz	63.14 kHz

The values in this table were calculated assuming that the fit value for the resistance is correct and that the resistor is perfectly non-inductive. The second assumption led to phase errors calculated as deviations from 0°.

In practice, the phase limit was always exceeded at a lower frequency than the magnitude limit.

Conclusions

In this Technical Note, Gamry Instruments presents a number of guidelines for accurate EIS measurements on low impedance cells. Galvanostatic cell control, a large AC current, and twisted-pair cell wiring are all important.

When these guidelines are followed, an EIS system equipped with a Gamry Instruments Reference 600 can accurately measure the impedance spectrum of a low inductance 1 mΩ resistor.

When the resistor spectrum is corrected by series subtraction of a low impedance copper surrogate, the accuracy of the measurement is extended to higher frequencies.

Some, or all, inductance remaining after spectrum subtraction may be real inductance in the resistor. Note that the 390 pH inductance value calculated in a series RL model fit is lower than the resistor family's claim of 0.5 to 5.0 nH inductance.

Assuming the 1 mΩ resistor that was used in this test is perfectly non-inductive, its impedance can be measured using a Reference 600 based EIS system out to 12.6 kHz.