Lithium-ion batteries have enjoyed widespread use in portable electronics for over two decades, and are increasingly relevant for electric vehicles as operational life and energy density continue to increase. The use of high voltage Li$_x$Mn$_{1-x}$O$_2$ (M = Ni, Mn, Co), or NMC (Li[Ni$_{0.42}$Mn$_{0.42}$Co$_{0.16}$]O$_2$), positive electrode materials improves the energy density of the cell, but difficulties arise in maintaining calendar life and coulombic efficiency, especially at voltages greater than 4.3 V. The internal impedance of a cell is closely associated with its health, so electrochemical impedance spectroscopy (EIS) is a powerful diagnostic technique. It is important to connect the features seen in EIS spectra with the correct internal processes occurring within a cell. A comprehensive circuit model for the impedance of a cell is needed to understand these processes.

Figure 1a shows capacity vs cycle number for two NMC442/graphite Li-ion pouch cells (240 mA/h). The high magnitude of the high frequency impedance is due to the two-wire measurement apparatus. The impedance of the wires contributes to the overall series resistance measured. The exact electrode composition in the studied cells was:

- Positive electrode: 95.4%:1.3%:1.1%:2.2% = active material : carbon black : PVDF Binder; negative electrode - 95.4%:1.1%:2.2% = active material : carbon black : CMC : SBR. The pouch cells used were so called 402035 size which were 40 mm long by 20 mm wide by 3.5 mm thick.

The positive electrode coating had a total (both coatings and current collector) thickness of 105 μm and was calendared to a density of 3.55 g/cm$^3$. The negative electrode coating had a total thickness of 110 μm and was calendared to a density of 1.55 g/cm$^3$. The positive electrode coating had an areal density of 16 mg/cm$^2$ (one side) and the negative electrode had an areal density of 9.5 mg/cm$^2$ (one side). Both the positive and negative electrodes were coated on both sides, and the electrodes were spirally wound yielding an active area of around 100 cm$^2$. One was cycled between 2.8 V and 4.4 V with a 24 h hold at 4.4 V, and the other was cycled between 2.8 and 4.5 V with a 24 h hold at 4.5 V (see Figure 1b). Additional information on the preparation of these cells is outlined by Nelson et al. The 4.5 V cell shows considerable capacity fade after 30 cycles, whereas the 4.4 V cell has good capacity retention for the first 80 cycles. Figure 1c shows the impedance spectra of the 4.4 V cell taken at 4.5 V. The shifting of the spectra is seen, accompanied by large growth of the diameter of the semicircle in the mid-frequency regime at 4.5 V. Figure 1 shows impedance spectra for the same cell taken at 3.8 V. The diameter of the semicircles are much smaller than in Figure 1e, indicating that the impedance growth at high voltage is largely reversible. However the diameters of the spectra are still increasing slowly with cycle number irreversibly. The spectra in Figures 1c–1f have inductive tails at high frequency due to the internal inductance of the measurement device used to collect the data.

It has been previously established by Chen et al. that this impedance growth exemplified by Figure 1 is mainly due to degradation of the positive electrode. To support this claim, Figure 2 plots symmetric cell impedance plots for LaPO$_4$-coated Li[Ni$_{0.42}$Mn$_{0.42}$Co$_{0.16}$]O$_2$/graphite (NMC442) pouch cells. Figure 2a shows the positive electrode symmetric cell impedance and Figure 2b shows the negative electrode impedance. The green stars signify the 1 Hz points. The positive electrode has greater impedance than the negative electrode by a full order of magnitude. Additionally, the positive electrode symmetric cells exhibit extremely high impedance at frequencies lower than 1 Hz, whereas the negative electrode symmetric cells exhibit much lower impedance at frequencies higher than 1 Hz. This indicates that the high impedance seen in Figure 1e is because of positive electrode effects, both due to the order of magnitude and the frequency regime in which it is found.

The exact cause for the reversible and irreversible increases in impedance at high voltage is still a matter of discussion. The work of Kerlau et al. postulates electrical contact resistances between the carbon black particles and the active particles in the cathode develop as the cell cycles. These additional resistances are claimed to cause a substantial increase in the diameter of the Nyquist spectra without significantly affecting the high frequency intercept. Metzger et al. claim anodic oxidation of carbon black in the cathode and ethylene carbonate in the electrolyte prevents the cell from operating close to 5 V. Metzger et al. also claim the oxidation of the carbon black leads to degradation of the electrode conduction path in the electrolyte. Nelson et al. suggest reversible impedance growth is caused by surface compounds that form on the cathode particles at high voltage and return to solution at low voltage, i.e. a dynamic solid electrolyte interphase (SEI). Reversible impedance growth was suggested to be primarily due to continued SEI growth on the positive electrode surface during cycling as well as electrolyte degradation.

The transmission line model (TLM) theory of the internal impedance of an electrode mimics the geometry and interfaces in the electrode. Figure 3b shows a simplified diagram of a cell positive electrode and Figure 3a shows a possible corresponding discrete
transmission line model. $R_e$ represents the electronic path resistance through the carbon black. $R_i$ represents the ionic path resistance through the electrolyte solution in the electrode pores. In this model, $R_e$ and $R_i$ have the same overall effect on the circuit. $C$ represents the capacitance of the SEI double layer that forms on the surface of the active particles as the cell cycles. $R_t$ represents the associated charge transfer resistance of this SEI layer. $R_c$ represents the contact resistance between the carbon black and the active particles, as outlined by Kerlau et al. in Figure 7b of their paper. Their model placed $R_c$ resistors in series with the RC pairs that represented the SEI of the active particles. The $R_e$ resistors in this TLM model have been added in the same place in order to make a direct comparison with their results. Further complexity can also be included such as a parallel RC pair to represent the contact impedance between the electrode and current collector as outlined by Atebamba et al., but that will not be included here. In addition, Warburg impedances can be added to mimic solid state diffusion, which affects the lowest frequency sections of the impedance spectra, but Warburg impedances will not be considered here. “Constant phase elements” which have questionable physical significance are often used to replace transmission...
Symmetric cell impedance for lanthanum phosphate coated NMC442/graphite Li-ion pouch cells. (a) Positive electrode impedance. (b) Negative electrode impedance. Impedance of the positive electrode is greater by an order of magnitude.

In this paper, constant phase elements are not used to try to maintain simplicity and physical reality.

Continuous transmission line models for cylindrical pores have already been developed and explored. While they are mathematically concise, they often assume constant resistivity and permittivity for the associated components in the model. As such, they cannot account for inhomogeneities in the positive electrode particles. In this study, a discrete transmission line model is solved using circuit dynamics for various circuit parameters. By systematically altering the circuit parameters and observing the changes in the impedance spectra, a guide to understanding changes in measured impedance spectra is developed.

Theoretical

The complex impedance of the transmission line model shown in Figure 3 was calculated both analytically and numerically via SPICE simulation. Figure 4 shows the process by which the transmission line was reduced from 5 links to 4 links using Y-Δ transforms. This process was executed repeatedly until the circuit was solved. In the homogeneous case, the value of each circuit component was constant from link to link. In the inhomogeneous case, each individual circuit element could have a unique value. The number of links in the TLM was chosen to be five for the following reasons:

SEM images of NMC positive electrode particles in Figure 9 of Li et al.’s publication show a particle diameter ranging between 15–20 μm. SEM images of NMC particles from commercial 18650 cells (Figure 7 in the paper by Burns et al.) show the particles to be about 15 μm. Commercial NMC positive electrodes are coated on both sides of an aluminum current collector which is normally about 10–15 μm thick. Gyenes et al. list a total positive electrode thickness of 105 μm in Table I of their paper which suggests a single side coating with a thickness of about 50 μm. NMC cells designed for energy applications may have thicker positive electrodes, perhaps up to as much as 100 μm. Positive electrode coatings between 50 and 100 μm thick are only about 4 to 10 particles “deep”. That is why a 5 link TLM was selected. Similar statements can be made for LiCoO₂ (LCO) and Li[Ni₀.₈Co₀.₁₅Al₀.₀₅]O₂ electrodes where large particles are used, but not for typical LiFePO₄ electrodes where much smaller particles are used and hundreds or thousands of links would be required.

The impedance spectra produced by the theoretical calculation were compared to SPICE simulations of the same circuit, and were

![Figure 3](image-url)
frequency in Figures 1e and 1f. During manufacturing of a Li-ion cell, the positive electrode material is calendared at high pressure to the current collector, minimising the resistance. Its contribution is evidently small compared to the mid-frequency impedance growth and has been omitted from the model for the sake of simplicity.

Results and Discussion

Figure 5 shows impedance spectra generated by the discrete TLM. The spectra were generated to roughly coincide with the measured data in Figures 1c and 1e. The red curve in Figure 5a corresponds to \( R_\pi = 0.26 \Omega \). \( R_\pi \) increases linearly for the intermediate spectra and ends at 0.32 \( \Omega \), represented by the blue spectrum. If \( R_\pi \) were held at 0.25 \( \Omega \) and \( R_s \) were allowed to vary between 0.26–0.32 \( \Omega \), the resultant spectra would be the same. This is reflected in the fact that \( R_\pi \) and \( R_s \) have identical effects on the circuit. As \( R_\pi \) increases, the diameters of the spectra increase slightly, but the dominant effect is an appreciable increase in the real part of the impedance intercept at high frequency. In the context of Figure 1a, this indicates that the electronic path or the ionic path resistance is increasing in the cell during the aggressive charge- hold-discharge cycling in Figure 1. A change in the charge transfer resistance is not necessary to explain the shift in the real part of the cell’s impedance. Figure 5b was generated similarly to Figure 5a with \( R_\pi \) increasing from 1.25 \( \Omega \) (red) to 1.5 \( \Omega \) (blue). Additionally, the red curve corresponds to \( R_s = 0.3 \Omega \) and the blue curve corresponds to \( R_s = 3.5 \Omega \). The black curves correspond to intermediate values of \( R_\pi \) and \( R_s \). An increase in the SEI resistance of the double layer was necessary to produce the large diameter growth between the red curve and the blue curve. Changing \( R_s \) alone was ineffective in shifting the high frequency intercept. This is consistent with the model outlined

\[
Z(\omega) = \frac{\left( R_s^4 + 9 R_s^3 R_\pi + 9 R_s^2 Z_{cl} + 22 R_s^2 R_\pi^2 + 66 R_s^2 R_\pi Z_{cl} + 28 R_s^2 Z_{cl}^2 + 120 R_s R_\pi Z_{cl}^3 + 28 R_\pi^2 Z_{cl}^3 + 15 R_\pi Z_{cl}^4 + 9 R_\pi ^2 Z_{cl}^4 + 28 R_\pi^3 Z_{cl}^4 + 35 R_\pi^2 Z_{cl}^5 + 20 R_\pi Z_{cl}^6 + 5 Z_{cl}^7 \right)}{1 + j \omega R_\pi C + R_\pi}
\]

where:

\[
Z_{cl} = \frac{R_\pi}{1 + j \omega R_s C + R_\pi}
\]

and \( j \) is the imaginary unit. For the inhomogeneous case, or in the case of more links, the impedance was solved procedurally using a computer algorithm written in Python. This TLM excludes the case of more links, the impedance was solved procedurally using a computer algorithm written in Python. The surface area of the aluminum current collector is much lower than the total surface area of the SEI on the active particles. Since the resultant capacitance is lower, any capacitive effect due to the metal substrate will be seen at high frequency. This offers an explanation for the small peak at high frequency in Figures 1e and 1f.
in Figure 3 because the capacitors have near-zero impedance at high frequency. This causes the SEI resistors to be in parallel with a short circuit at high frequency, rendering them ineffective. As a result, the position of the high frequency intercept is not affected by changes in the SEI resistance. This indicates that the dominant process causing the shift in the high frequency intercept was still present in the cell when Rs was doubled to 2 Ω. An additional effect causing the diameter to increase accordingly. Increasing Rs has a small effect on the spectrum diameter because current in the circuit favors certain charge transfer paths, specifically ones that minimize transit through the increased resistance of the electronic path resistors. Because the bulk of the current travels through fewer charge transfer pathways which are in parallel, the charge transfer resistance increases. This is analogous to the fact that 2 resistors in parallel constitute a more resistive system than 5 resistors of the same value in parallel due to the reduction in electronic pathways. In the limit of large Rs, all the current will travel through the closest charge transfer pathway. Thus, if Rs is held constant and Rc increased, the total charge transfer resistance of the circuit (Rct) will asymptotically approach Rc. The presence of contact resistances has no effect on the spectrum diameter, because it is present in the circuit at both low and high frequency, and it does not increase the conductivity of some electronic pathways over others. The effect of each of these components on the impedance of the TLM is summarized in Figure 7. Figure 7a plots total charge transfer resistance (the spectrum diameter or Rct) vs. Rs, Rc, Re, and Ri. The base parameters of the circuit were Rs = 1 Ω, Rc = 0.25 Ω and C = 1 F. The order of magnitude of these values was motivated by empirical observation of the experimental resistance and frequency data in Figure 1. As one parameter varied, the others in each graph were held constant. The effect of Rs (shown in blue) is the most significant. Changes in Rc or Re have a small effect on Rct and Ri has virtually no effect at all. This indicates that a large increase in the spectrum diameter, as seen in Figure 1e for example, must be attributed to an increase in the charge transfer resistance of the active particles in the positive electrode. Figure 7b shows the position of the high frequency intercept vs. Rs, Rc, Re, and Ri. The base parameters are the same as in Figure 7a. Changes in Rc have no effect on the high frequency intercept as previously discussed. However, Rc, Re, and Ri can all potentially contribute to the shift on the real axis.

A closer examination of the individual effects of each circuit component was conducted. Figure 6a shows the transmission line model used to examine the effects of different circuit elements. Figure 6b shows Nyquist plots for impedance spectra. The base values for the circuit parameters were Rs = Rs = 1 Ω, Rc = 0.25 Ω and C = 1 F. The Nyquist spectrum of the circuit with these parameters is shown in black. The blue Nyquist curves represent the impedance of the TLM when Rs was doubled to 2 Ω and quadrupled to 4 Ω respectively with all other parameters held constant. The red impedance spectra were produced by increasing Rs or Rc to 0.5 Ω and 1 Ω with Rs = 1 Ω and all other parameters held constant. The spectra shown in green represent Rs being increased to 2 Ω and 4 Ω with all other parameters held constant. Rc has the most significant effect on the diameter of the spectra. An increase in Rs or Rc also increased the spectrum diameter, but it was also accompanied by a significant increase in the high frequency resistance. A change in Rs had no effect on the spectrum diameter. However, it caused an appreciable increase in the real part of the high frequency impedance. This is in disagreement with the hypothesis of Kerlund et al., who make the spurious claim that contact resistances have no effect on the high frequency intercept, but significantly increase the spectrum diameter.

To understand the individual effects of each resistor element on the spectrum diameter, one must examine the high and low frequency intercepts in the context of DC resistance of the TLM. The value of the low frequency intercept is the DC resistance of the TLM if the capacitors were removed from the circuit. The value of the high frequency intercept is the DC resistance of the TLM if the capacitors were replaced with short circuits. Because a short circuit in parallel with a resistor is equivalent to a short circuit, this is equivalent to shorting the charge transfer resistors and removing the capacitors. In this context, it is clear that increasing Rs in the TLM will have the most significant effect on the spectrum diameter. Increasing Rs alone cannot cause the high frequency intercept to shift, but it will cause the value of the low frequency intercept to increase because the DC resistance of the TLM is now larger. This increases the distance between the two intercepts and causes the diameter of the spectrum to increase accordingly. Increasing Rs has a small effect on the spectrum diameter because current in the circuit favors certain charge transfer paths, specifically ones that minimize transit through the increased resistance of the electronic path resistors. Because the bulk of the current travels through fewer charge transfer pathways which are in parallel, the charge transfer resistance increases. This is analogous to the fact that 2 resistors in parallel constitute a more resistive system than 5 resistors of the same value in parallel due to the reduction in electronic pathways. In the limit of large Rs, all the current will travel through the closest charge transfer pathway. Thus, if Rs is held constant and Rc increased, the total charge transfer resistance of the circuit (Rct) will asymptotically approach Rc. The presence of contact resistances has no effect on the spectrum diameter, because it is present in the circuit at both low and high frequency, and it does not increase the conductivity of some electronic pathways over others. The effect of each of these components on the impedance of the TLM is summarized in Figure 7. Figure 7a plots total charge transfer resistance (the spectrum diameter or Rct) vs. Rs, Rc, Re, and Ri. The base parameters of the circuit were Rs = Rs = 1 Ω, Rc = 0.25 Ω and C = 1 F. The order of magnitude of these values was motivated by empirical observation of the experimental resistance and frequency data in Figure 1. As one parameter varied, the others in each graph were held constant. The effect of Rs (shown in blue) is the most significant. Changes in Rc or Re have a small effect on Rct and Ri has virtually no effect at all. This indicates that a large increase in the spectrum diameter, as seen in Figure 1e for example, must be attributed to an increase in the charge transfer resistance of the active particles in the positive electrode. Figure 7b shows the position of the high frequency intercept vs. Rs, Rc, Re, and Ri. The base parameters are the same as in Figure 7a. Changes in Rc have no effect on the high frequency intercept as previously discussed. However, Rc, Re, and Ri can all potentially contribute to the shift on the real axis.

Figure 6. (a) Discrete TLM circuit used in simulation. (b) Assorted Nyquist plots examining changes in Rct (the diameter of the spectrum). The parameters written in black correspond to the black curve. The blue, green and red curves correspond to the increase of Rs, Rc and Rs or Rc, respectively, with all other parameters held constant. Changing Rs has the largest effect on Rct.

Figure 7. (a) Rct (the spectrum diameter) plotted vs Rs, Rc, Re or Ri. (b) Position of the high frequency intercept plotted vs Rs, Rc, Re and Ri. Rs has no effect on Rct, while Rs and Rc have a moderate effect that is asymptotically bounded and Rs has a large effect.
Inhomogeneities in $R_c$ and $C$ create a distribution in $RC$ times for each charge transfer pathway. This can cause a “spreading” of the high and low frequency intercepts without a proportional change in the peak position, or it can cause the peak position to depress without a proportional change in the charge transfer resistance. Figure 9a shows the variety of spectrum shapes that can result from inhomogeneity in the double layer capacitance of the active particles. The TLM circuit parameters are $R_s = 0.5 \ \Omega$, $R_c = R_t = 0.25 \ \Omega$ and $R_i = 0.5 \ \Omega$. 100,000 trials were conducted where each SEI capacitor was assigned a random value between 0.1–0.4 F. The red curve is a perfect semicircle. The region highlighted in black is the space that the 100,000 Nyquist spectra occupied, and two example spectra have been plotted in green. A “depression coefficient” is defined to quantify the degree of “flattening” of the Nyquist spectrum: 0% denotes a straight line along the real axis and 100% represents a perfect semicircle, where the height and width of the Nyquist spectrum are used for the calculation of the depression coefficient. Figure 9b shows a histogram of the depression coefficients for the 100,000 trials conducted. Spectra as flat as 76% were observed, with the most common depression being close to 90%. Figure 9c shows a similar 100,000 trial graph where $R_s = R_i = 0.25 \ \Omega$, $R_c = R_t = 0.5 \ \Omega$, $C = 1 \ \text{F}$ and the charge transfer resistors were each assigned a random value between 0.4–1.0 $\Omega$. The upper red curve and the lower red curve represent the $R_c$ arrangements that gave the highest and lowest $R_{ct}$ respectively. The black region is the space that the intermediate Nyquist spectra could be near semicircular, or asymmetric and depressed.
Figure 9. (a) Range of possible impedance spectra that result from randomized capacitances between 0.1–0.4 F. Example spectra are plotted in green. (b) Histogram of spectrum depression for 100,000 trials. (c) Range of possible impedance spectra that result from randomized SEI resistance between 0.4–1.0 Ω. Red curves represent smallest and largest $R_{ct}$ produced by these trials. (d) Histogram of spectrum depression for data in 8c. (e) Randomized capacitances between 0.1–2.0 F with the same format as 8a. (f) Histogram of spectrum depression for data in 8e. (g) $R_s = 0.05–1.0$ Ω with same format as 8c. (h) Histogram of depressions for data in 8g.

Figure 9d shows a histogram of the spectra depression for all 100,000 trials. Depression is evenly centered close to 89%. In Figure 9e, the range of capacitances was increased to a factor of 20. The black region terminates at a significantly lower value of $-\text{Im}\{Z\}$, and the example curves in green show that the spectra can exhibit large asymmetries and multiple peaks. Figure 9f plots the spectrum depressions for this trial. Spectra that reached depression under 60% were observed. In Figure 8g the range of $R_s$ was increased to a factor of 20. Depression down to 80% was observed in Figure 8h. The purpose of this figure is to explain why real impedance spectra can have asymmetries and can be measurably flatter than perfect semicircles. This effect can be compounded further if the RC time constant of the SEI in the negative electrode differs moderately from that of the positive electrode. The two peaks will overlap and resemble a flattened spectrum.

**Conclusions**

A discrete transmission line model for the impedance of a Li-ion positive electrode was explored. The impedance of the TLM circuit was solved analytically using Y-$\Delta$ transforms and the solutions were verified using SPICE. The TLM model was applied to example data that showed large growth in $R_{ct}$ at high voltage during aggressive...
charge-hold-discharge cycling. It was found that in order to increase the real resistance at high frequency, any of \( R_e \), \( R_c \) or \( R_s \) had to increase. However, only \( R_c \) could be responsible for the large increase in \( R_c \). It was found that any effect other than changing \( R_c \) that was able to change the total charge transfer resistance was asymptotically limited by \( R_c \). This suggests that high voltage impedance growth is due to continued SEI growth on the active particles, or the presence of surface species at high voltage.

An inhomogeneous set of electronic path resistances increased the high and low frequency resistance, but reduced the total charge transfer resistance of the circuit. An inhomogeneous set of contact resistances was able to cause an increase in the spectrum diameter, but it was limited to the value of \( \Delta R \) and could not explain high voltage impedance growth seen in aggressively cycled real cells. A range of random SEI capacitance/resistance values were used to simulate surface in-homogeneity in the positive electrode. The simulations produced “flattened” and asymmetric spectra in comparison to a semi-circle. This agreed qualitatively with the flat and asymmetric spectra seen in impedance measurements of real cells.

These simulations and comparisons to real data suggest avenues for improving the positive electrodes in NMC/graphite cells destined for high voltage operation:

1) The electrode/electrolyte interface must be improved by changes to the electrolyte or the electrode surface.
2) The integrity of both the electronic path and the ionic path must be maintained. The conducting diluent cannot be oxidized and pores cannot be blocked by electrolyte decomposition products.
3) The current collector/electrode interface must also be maintained.

Based on the work shown here, dealing with the electrode/electrolyte interface is most critical, but all of 1), 2) and 3) must be solved to guarantee success.

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