Key benefits and principles of isothermal and adiabatic calorimetry

Key benefits and principles of Isothermal and Adiabatic calorimetry

Joseph Willmot – 16/12/2020

Shlomit Dery (00:11):

Everybody welcome and happy Hanukkah. We are happy to host a Dr. Golik Academy. Today we are in the final lecture of the energy week webinars. I'm exciting to host Joseph Willmot, application leader from HEL Group, our partners. If you have any question during the webinar, feel free to write it in the chat and we will answer or respond in the end or later on. And thank you for joining. Enjoy. Joseph.

Joe Willmot (00:44):

Thank you very much. So yes, good afternoon. I hope you have all been enjoying the previous presentations that have been happening throughout the week. I've found them quite enjoyable. And so the final webinar that we'll be going over today, we'll be discussing the key benefits and principles of isothermal and adiabatic calorimetry, and essentially how they will help aid in the design of better batteries and better cells. This presentation should probably take around about 40 or so minutes, so there will be plenty of time at the end of the presentation to go through some Q&A questions. If you do have any questions, please enter them into the chat as we go, and I'll answer them near the end.

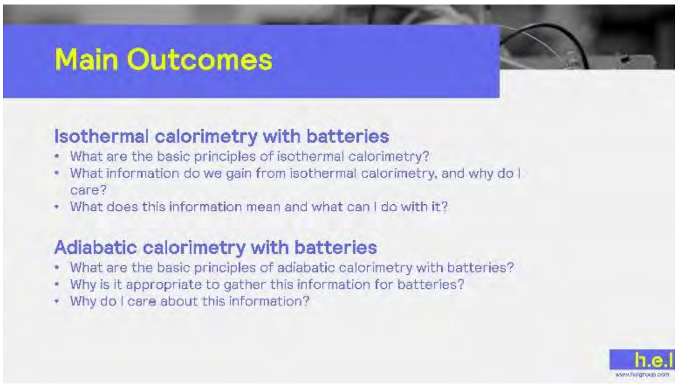
Presenters

Joseph Willmot – Application Leader

Spent 3 years as a Project Manager, which allowed him insight into the functionality of the various equipment H.E.L produces. He is now utilising this knowledge as Application Leader, where he is now looking at customers usage of the equipment, and helps them find the best solution for their application

Joe Willmot (01:34):

So some brief introductions. There we go. First of all, who am I? My name is Joseph Willmot. I'm the application leader here at HEL Group. I spent a couple of years as a project manager, which was building and developing the equipment. Whereas now I'm taking more of a application focus, looking at what our customers use our equipment for, and our battery testing equipment for, to help them get the most out of their equipment and help them with their actual application of the science that they're actually trying to achieve.



Main Outcomes

Isothermal calorimetry with batteries

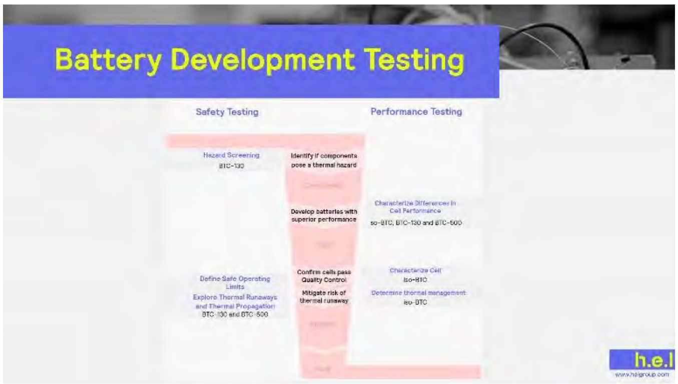
- What are the basic principles of isothermal calorimetry?
- What information do we gain from isothermal calorimetry, and why do I care?
- What does this information mean and what can I do with it?

Adiabatic calorimetry with batteries

- What are the basic principles of adiabatic calorimetry with batteries?
- Why is it appropriate to gather this information for batteries?
- Why do I care about this information?

Joe Willmot (02:16):

So what are going to be the main outcomes from today? There'll be two main areas that we're going to cover. One will be the performance battery testing in the isothermal calorimetry, and we'll also be looking at safety testing with adiabatic calorimetry. So with isothermal calorimetry, we'll be understanding the basic principles of isothermal calorimetry, what information we gain and what does this information mean, and what you can use this information for. For adiabatic and for safety testing, we'll go over what the basic principles of adiabatic calorimetry are, and how do we gather this information from batteries, and what's appropriate for us to gather from batteries? And then again, why do I care about this information? Just because I know what the maximum temperature of operation of my battery is, how does that dictate? Just because I've got that information, what do I then do with then? How do I then process it?



Battery Development Testing

Joe Willmot (03:12):

So let's think about battery development testing overall, all together. As I've said, we've got the safety testing side of things and we've got the performance testing side of things. With safety tests, and we structured this as a funnel where we've got lots of potential options and lots of potential different compounds, right at the start of the funnel. And as we progress through the cell into module, into the pack development, the number of variants are going to be reduced, but the level of detail and the quantification of data is going to increase. Now, we've pictured this as a funnel, but we do appreciate that multiple different sections could be happening at the same time.

Joe Willmot (03:55):

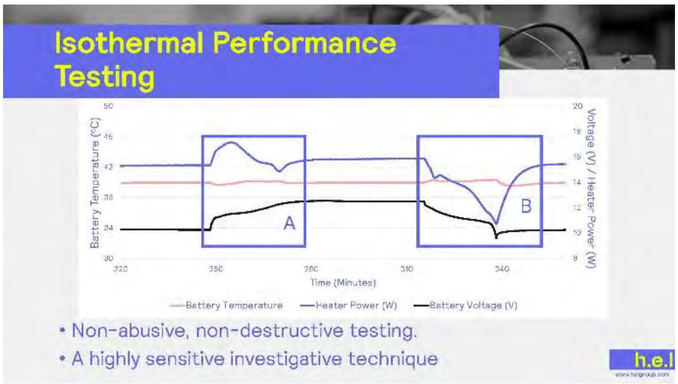
Right at the top of the funnel, you're going to have your potential components that you want to work with. You're going to have your different electrolytes. You're going to have your different individual components that you're going to be building and putting together into yourself. And you're going to want to make sure that they're appropriate to use and safe to use. So that would be the very first kind of hazard screening that you're going to be entering in. As we go through into developing it into a cell, you then want to characterize the performance of the cell, make sure it's up to specification, make sure it's up to what you're wanting to achieve from the cell. And then finally, as we go down into the module and we start putting the cells together, we're going to want to make sure that our thermal management strategies are appropriate for the entire battery pack, as well as defining the safe operating limits and designing the entire system around the battery pack to ensure the conditions that you've designed the battery to be used in, are maintained to ensure that the property is safe to use.

Joe Willmot (04:55):

So we'll be going over both of these. To the safety testing, I'll come to that second. So we'll start off by looking at the isothermal calorimetry and the performance testing side of things. And voilà.

Performance Testing

Isothermal Calorimetry



Isothermal Performance Testing

- Non-abusive, non-destructive testing.
- A highly sensitive investigative technique

Joe Willmot (05:09):

So let's first of all, all get on to the same page. Let's all consider what isothermal calorimetry is and how it is going to be useful for us to understand, and help characterize these cells that we're going to be testing. Isothermal calorimetry, is a non-abusive, non-destructive testing and it's in a highly sensitive investigative technique. It involves holding your sample, in this case our battery, at a constant temperature and measuring the thermal duty that the battery is going to undergo under operation. This is commonly referred to as the samples heat flow. In the chemistry world, that would be mixing of two compounds together, two chemicals together, measuring the energy released from them to understand what reaction has taken place.

Joe Willmot (05:57):

For batteries, we know what reaction has taken place. For lithium-iron batteries, we want the lithiation and delithiation of your anodes and cathodes. So we know the reaction that's taking place, but we want to quantify the energy to ensure that we're capturing the entire energy of the discharge and charging of our batteries, or cells, or battery packs, et cetera. So because of that, we want to eliminate heat losses. We want to eliminate any variants of the cell and we want to hold it isothermally. If we hold it isothermally, the heat losses of the cell aren't going to increase or decrease, which means we don't have to worry about compensating for that, by saying different wattages at different temperatures and so on.

Joe Willmot (06:46):

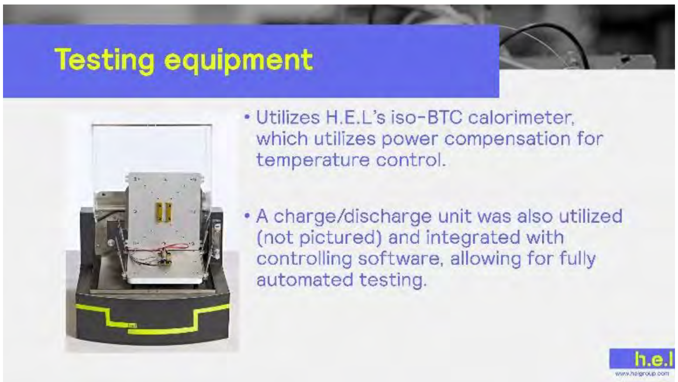
So we start by holding our battery at a very stable temperature. In this particular case as you can see, we're holding it at 40 degrees C, as is the pink line on the left-hand axis. We then have a controlling heater that's wrapped around the outside of the cell and undergoes power compensation to maintain the battery's temperature. We have two cooling plates that are above and below the battery, which provide the cooling side of things. And then we use the heater that's wrapped around the battery to input heat and perform the temperature management system of the battery. And this is commonly referred to as power compensation temperature control.

Joe Willmot (07:29):

With that, we've got this calibrator teeter that's wrapped around the outside. So whenever we're performing a thermally dynamic duty on the battery, such as charging or discharging it, by however much we need to increase or decrease the power to that heater, we can therefore quantify the amount of thermal duty that the battery is undergoing. So on this left-hand, in section A, this is where we're charging the battery. You can see on the right-hand axis, we've got, sorry, oh, yes, on our right-hand axis we've got our batteries in volts, and we've also got the heat power on the right-hand axis. So you can see that we're increasing our volts, we're charging our battery. You can see that the heater power has had to increase to maintain isothermal conditions. Because of that, we're putting energy in so we know that our charging process here is endothermic. And the exact same opposite happens on the other side. We're reducing our heat power, we're discharging the cell, therefore note that that is an exothermic reaction.

Joe Willmot (08:32):

Now that's obviously very simple. And if we compare the peaks and troughs of the heater power in comparison to the baseline, up against it's standardized isothermal conditions when the battery wasn't undergoing any charging or discharging, we can therefore quantify the amount of heat released from the cell as the cells heat flow. So that's essentially the basics of isothermal calorimetry on batteries.



Testing equipment

- Utilizes H.E.L's iso-BTC calorimeter, which utilizes power compensation for temperature control.
- A charge/discharge unit was also utilized (not pictured) and integrated with controlling software, allowing for fully automated testing.

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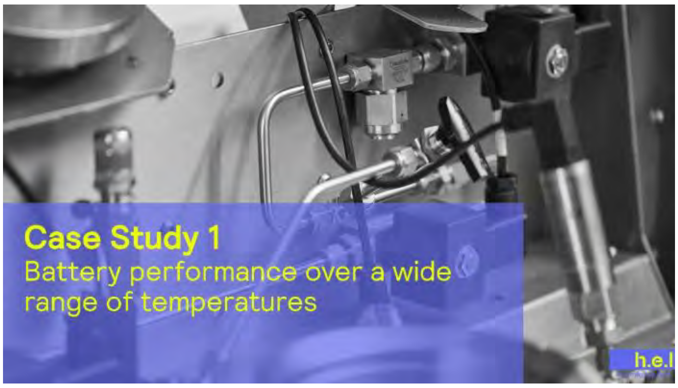
As I said, the testing equipment that we use to characterize this is the Iso-BTC. It utilizes power compensation and we normally couple this up with a charge and discharging unit. But I don't want to focus on the Iso-BTC, that's not what we're here for today.

Joe Willmot (09:15):

Let's consider how we would use, this isothermal calorimetry to help characterize some of the batteries.



Case Study 1

Battery performance over a wide range of temperatures

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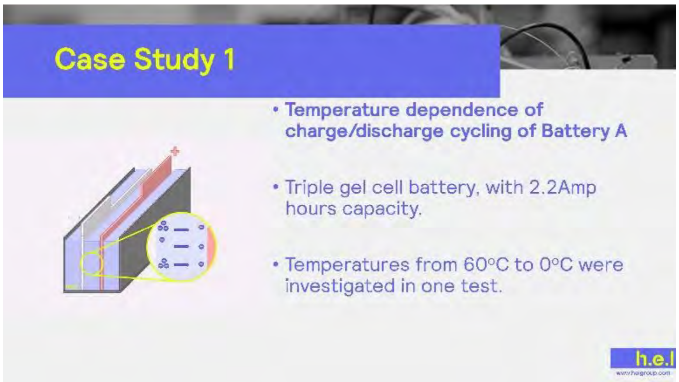

Case Study 1

- **Temperature dependence of charge/discharge cycling of Battery A**
- Triple gel cell battery, with 2.2Amp hours capacity.
- Temperatures from 60°C to 0°C were investigated in one test.

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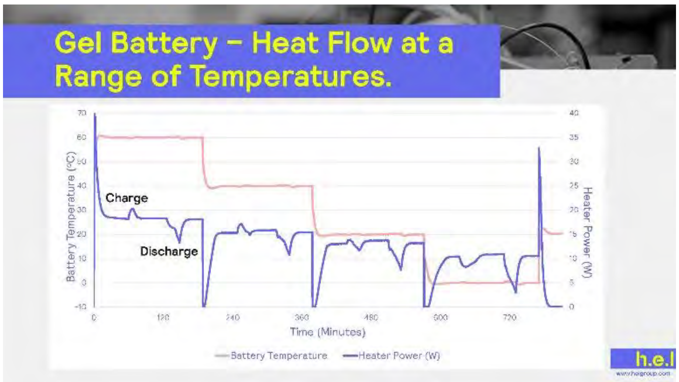
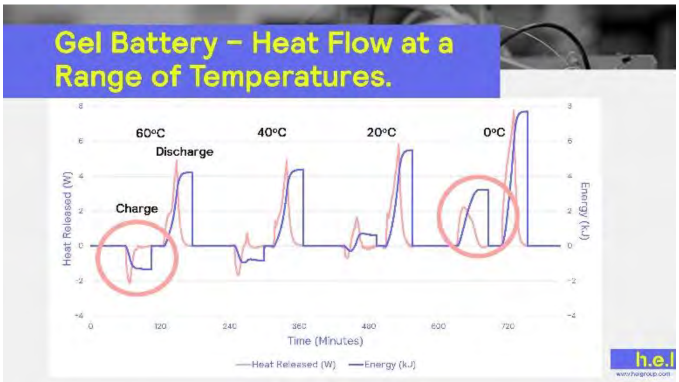
So the first example that I'll walk through today is, we wanted to characterize a cell over a wide range of temperatures to see how it's heat flow, how it's thermal duty changed over variety of temperatures. For commercial reasons, I can't tell you what this battery was, but I can describe it to you as a triple cell battery with a 2.2 amp hours capacity to it. We charged and discharged the battery from temperatures from 60 degrees C down to zero degrees C. And we managed to complete this within the Iso-BTC with just one simple test.



Gel Battery – Heat Flow at a Range of Temperatures.

Joe Willmot (10:09):

So here we can see the battery temperature in pink, on the left-hand axis and the heat power in blue, on the right-hand axis. You can quite clearly see the peaks and troughs that come from the charge and discharge when we were charging and discharging the cell. And you can see that already, we've seen some transitional behavior start to occur, especially with the charging.

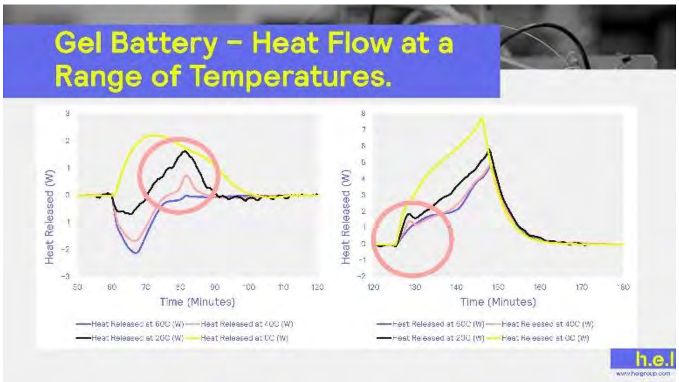


Gel Battery – Heat Flow at a Range of Temperatures.

If we compare the current heat power against the baseline, we therefore get the heat flow of the cell. And this is what the heat released in pink is in this graph here. We then integrate the area underneath these peaks to accumulate the total energy of either the charging or discharging process.

Joe Willmot (10:57):

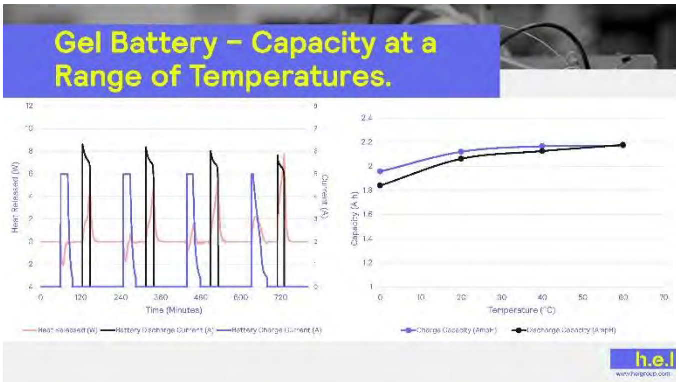
So up at 60 degrees C, we can see the charge is entirely an endothermic process. We can see that it's absorbing energy from the Iso-BTC. To maintain its isothermal temperatures, it's absorbing energy, we're having to input more energy to maintain isothermal conditions, so it's an endothermic process. However, at zero degrees C, we can start to see that it's actually becoming an exothermic process. On the right-hand side, we can see that the charging is now entirely an exothermic process rather than endothermic. So we can already see that there's obviously some internal, the battery is having to compensate for the temperature that it's undergoing. So here we have our endothermic process and here we have our exothermic process. So that's already quite interesting to show how the changing of temperature of the battery has ended up changing a process from an entirely an endothermic to entirely an exothermic.



Gel Battery – Heat Flow at a Range of Temperatures.

Joe Willmot (12:00):

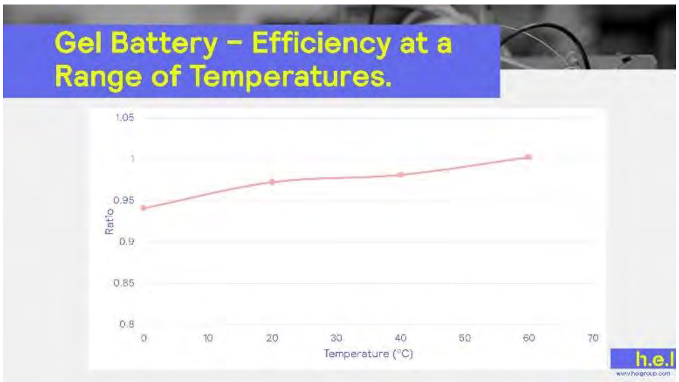
If we break down the individual charge and discharge profiles they're respective temperature in a bit more detail, we get these plots here. The charging is on the left-hand graft and the discharging is on the right-hand graph. So from that transition from endothermic to exothermic, on the discharging profile, we're getting a continuously, a greater exothermic process each and every time that we're dropping the temperature. We also have some slight peaks. We have a slight exothermic peak at the end of the charging process and we also have a small exothermic peak at the beginning of the discharging process here as well. And these correlated to actually the exact same voltage of the battery, which is quite interesting to try and understand of why those voltages caused exothermic peaks to occur. And obviously if we knew more about the internal, if I could talk to you about more about the internal working of the cell, I could try and explain to you why that occurred. But obviously as a cell manufacturer, they would be considering changing components to potentially remove these peaks or reduce these peaks to ensure that the exotherms were either reduced or completely eliminated.



Gel Battery – Capacity at a Range of Temperatures.

Joe Willmot (13:35):

So another way to use this information is if we actually look at the capacity of the battery over a range of these temperatures. If we integrate the current during the charging and discharging, we therefore get the capacity of the individual charge and discharge. And we can then plot these against each other on the right-hand axis. And you can see that the charge capacity at zero degrees C was just below two, but the charge capacity up at 60 degrees C was just below 2.2. So we're getting a greater capacity of the cell at a higher temperature. If we look at the ratio between these two, we get the efficiency of the cell.

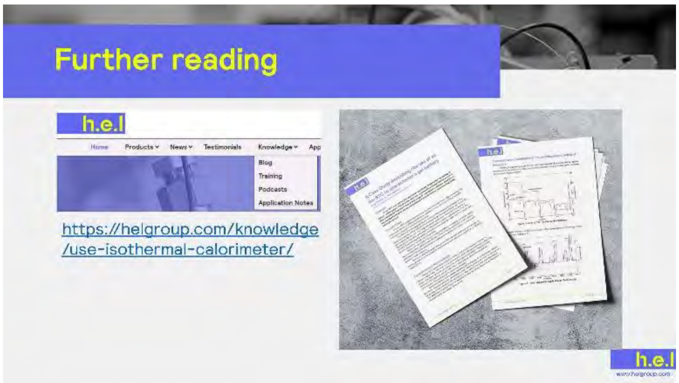


Gel Battery – Efficiency at a Range of Temperatures.

Then if we plot that against the temperature, we can see that the efficiency of the cell up at 60 degrees C is slightly above one. So with experimental error, we would obviously say that it's essentially all the energy that we're putting into the cell in charging, is being dissipated in discharging as well. Whereas if we look down at zero degrees C, we can see that the efficiency of the cell is dropping as we drop in temperature. Again, that's very useful information to know from a development standpoint.

Joe Willmot (14:52):

You might be looking into continuously running these cells at 60 degrees C to ensure that you have a highest efficiency possible, or perhaps you could change some internal components to ensure that it's more optimal operating temperature, that it's efficiency is nearer one at lower temperatures. If let's say you're designing a battery for use at lower temperatures, you would want to try and increase its efficiency at those temperatures that you've specified. So that's an example for a triple gel cell battery.



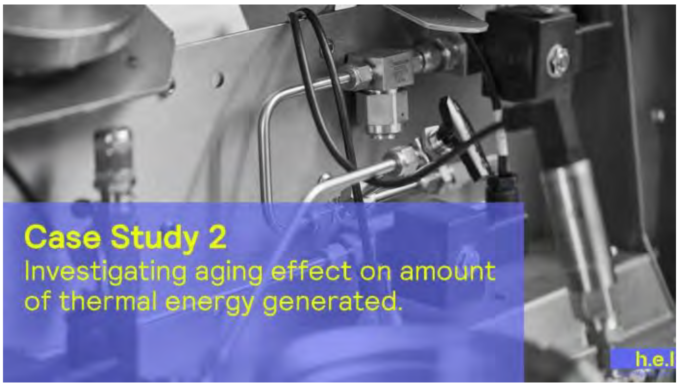
Further reading

https://helgroup.com/knowledge/use-isothermal-calorimeter/

Case Study: How to use an isothermal calorimeter to characterize a gel battery

Joe Willmot (15:28):

We've broken down a lot more characteristics such as looking at those voltages where we saw those exothermic speeds in a application note that is available on our website for free download. So please do go away and read up a little bit more detail about how the temperature affected the performance of this battery.

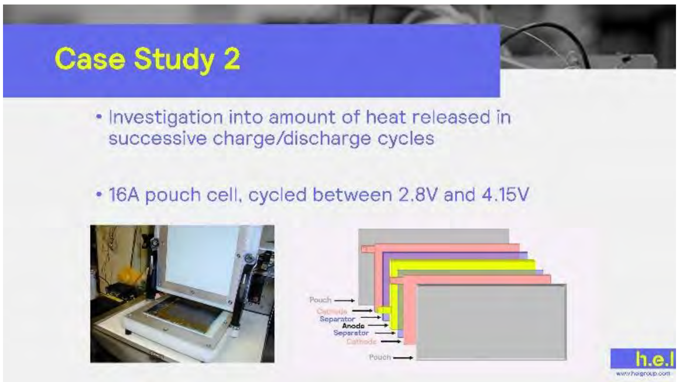


Case Study 2

Investigating aging effect on amount of thermal energy generated.

Joe Willmot (15:50):

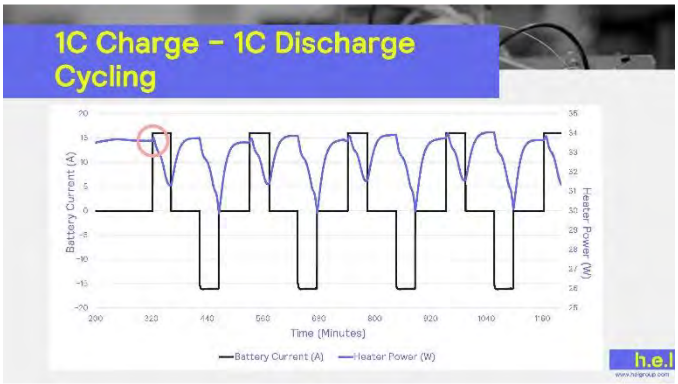
The second case study I'll talk to you about for isothermal is investigating the effects of aging that we had on the amount of thermal duty that the cell provided.



Case Study 2

- Investigation into amount of heat released in successive charge/discharge cycles
- 16A pouch cell, cycled between 2.8V and 4.15V

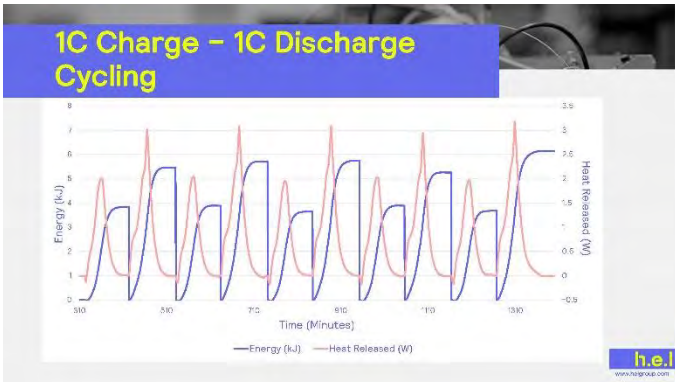
This was a pouch cell that was placed within the Iso-BTC. It had a C rate of 16 amps, meaning that it was rated by the manufacturer to be challenged at 16 amps and dissipated at 16 amps. But as I walk you through this, you'll obviously be able to see that a similar process, we wouldn't necessarily need to know what the C rate, what the rated charging and discharging rate of the pouch cell was. We could do this in reverse to find out what the actual safe charging and discharging rate was.



1C Charge – 1C Discharge Cycling

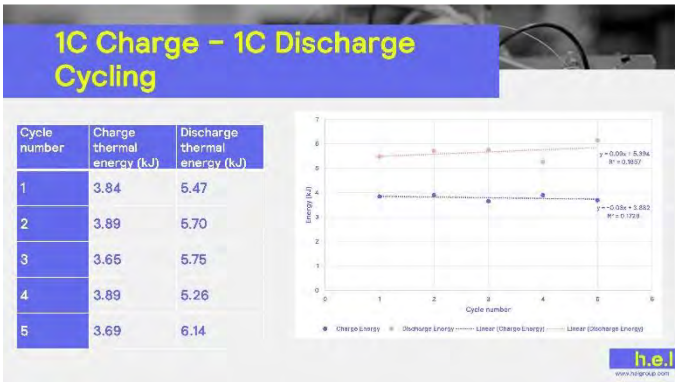
Joe Willmot (16:44):

So we first of all wanted to check that the C rate stated was appropriate and we cycled the unit. We cycled the charge and discharging of this pouch at a 1C charge rate and a 1C discharging rate between 2.85 and 4.14 volts, I believe it was. So you can see here that again, for both of these processes, heater power on our right-hand axis here in blue, is continuously having to drop, meaning that when we integrate this area under here and we see the heat flow of the battery, we know that this cell, sorry, not battery. We know that this cell is continuously being exothermic for charging and discharging. We can say in episodes like endothermic peak, right at the beginning of the charging process and it's repeatable, but it's mainly dominated by the exotherm of the rest of the charge.


1C Charge – 1C Discharge Cycling

Joe Willmot (17:44):

So again, we look at the heat released from the battery, we look at the heat flow of the cell, we integrate this to look at the total energy, the total thermal energy provided from this cell during it's 1C charging and 1C discharge.

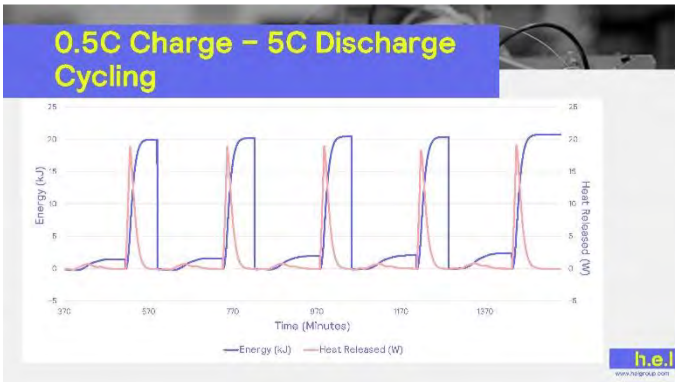

1C Charge – 1C Discharge Cycling

Cycle number	Charge thermal energy (kJ)	Discharge thermal energy (kJ)
1	3.84	5.47
2	3.89	5.70
3	3.65	5.75
4	3.89	5.26
5	3.69	6.14

If we then compare all of those integrated energies against each other, we can see that over this only short period of five cycles, there's not a massive amount of change in the battery's, in the cell's performance. We can see that the charge thermal energy essentially stays the same with the discharge level energy slightly increasing. And what this shows us is that the 1C charge and 1C discharge quoted for the cell is actually fairly appropriate. The performance of the cell hasn't changed dramatically. Now different temperatures could be investigated to see whether that accelerated or decelerated the aging effects of the cell. And obviously I wholly appreciate that this was only done over five cycles, which is a very short sampling window, but obviously this could be done over a much longer period to increase the aging of the cell, to see how it performs over time.

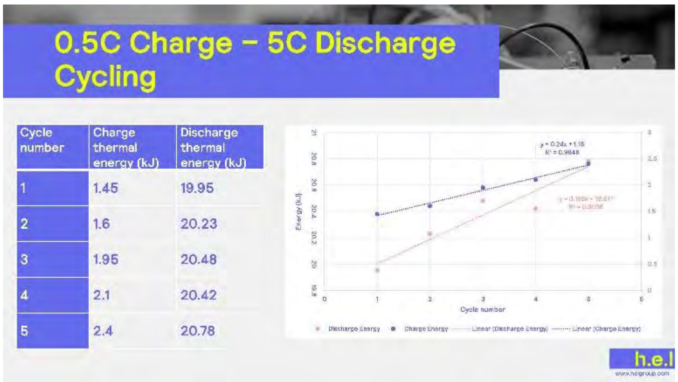
Joe Willmot (19:04):

What could also be done and what we did, is that we changed the discharging rate and we changed the charging rate. We changed the charging rate to nor 0.5 C, so we're charging it eight amps, and then we changed the discharge rate to be 5C, so we're now drawing 80 amps out from this pouch cell. So that's quite significant, we're really stressing the unit and stressing the cell to really try and get the discharge going as quickly as we safely can to really stress the cell, and to see how well it will perform.



0.5C Charge – 5C Discharge Cycling

From this, you can see that the energy produced during the charging periods is significantly reduced and the thermal energy released during the discharging period is significantly increased. We can see that we're now up at around about 20 kilojoules of thermal energy being out footage from the cell during the discharge. Which makes complete sense, we're really pushing this cell far beyond what the manufacturer rated it for, just to really accelerate the rate of aging that we're observing on this cell.



0.5C Charge – 5C Discharge Cycling

Cycle number	Charge thermal energy (kJ)	Discharge thermal energy (kJ)
1	1.45	19.95
2	1.6	20.23
3	1.95	20.48
4	2.1	20.42
5	2.4	20.78

Joe Willmot (20:14):

Unless your eyes are better than me, it's still very hard to compare all of these energy values. So on the table format, again, we see quite clearly a trend here. We can see that not only the amount of thermal energy given off from this cell has increased for both the charging and the discharging of the cell, which shows that this is quite rapidly aging the cell, potentially to a point where it becomes, dangerous to use or something like that. That the potential battery temperature control system designed for the cell would no longer be appropriate. So with this, as I said, you could quite clearly see how you could go backwards and not know what the safe charging and discharging rate of a cell is.

Joe Willmot (21:08):

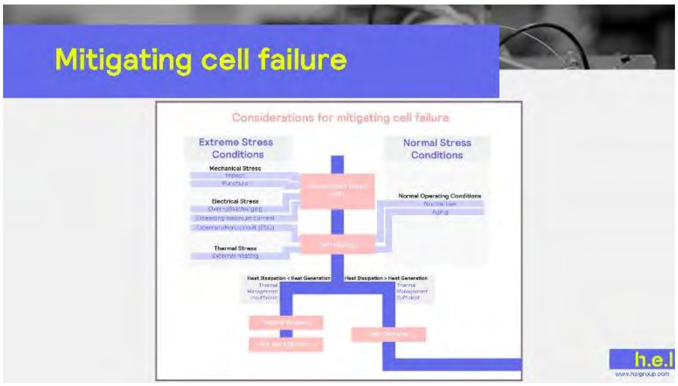
You could perform similar studies such as this with isothermal calorimetry, to define what your charging rates and discharging rates are, of your cells. And from that, obviously we're getting a thermal duty that's coming out from the cell during use, which could be directly applied to your thermal management system, for you to understand how much cooling is required to keep the cell at its potential optimal efficiency, to ensure that when you charge and discharge the cell, you get 100% of your energy back that you are thinking of.

Safety Testing
Adiabatic Calorimetry

So that's how isothermal calorimetry can, just some of the uses of it. There are multiple more examples, which I do go into elsewhere. And I'll give you a link to that a little bit later on.

Joe Willmot (22:02):

Next, let's consider some of the safety testing. Some of the safety testing that would be happening in parallel to this performance and characterization testing.



Mitigating cell failure

Considerations for mitigating cell failure

Safety testing, essentially, we're trying to mitigate cell failure. We're trying to ensure that when we have a cell, that it has the widest range of use available to it. So the widest range of voltages and charging rates and discharging rates and the widest range of temperatures as well. Now there will always be a play between performance and safety, as well as costs and other factors the people are going to need to consider when developing batteries, but safety really should be a very high priority, very important one.

Joe Willmot (22:52):

How do we test how safe a cell is? Well, the easiest way is to see how it performs under extreme stress conditions. We have mechanical testing, where we have impact and puncture testings. We've got electrical testing where we take the cell far beyond what it would ever see in its normal lifetime. And then we've also got thermal stress testing, where adiabatic calorimetry really starts to play its part. We've obviously also got normal stress testing conditions, which will be useful to know so you can determine the most appropriate lifetime of a battery.

Joe Willmot (23:34):

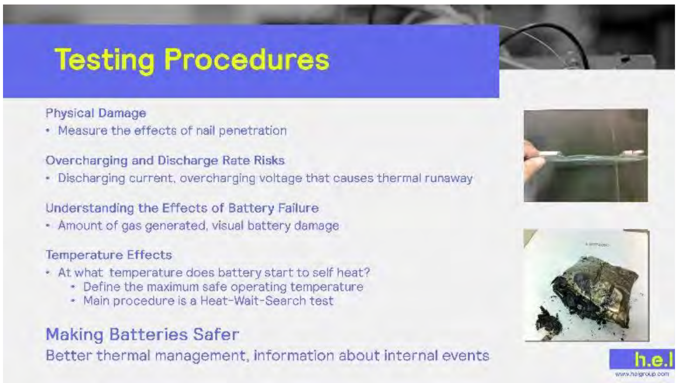
Now all of these kind of external stresses caused on a cell will cause some form of an internal short, and these will normally lead to some form of self-heating the cell. If the heat dissipated is greater, so if we're losing the heat quicker than it's accumulating heat, then although the cell might suffer subpar performance and eventually be damaged beyond use, essentially we have to the bottom rate a safe outcome. The cell isn't going into a thermal runaway, it's dissipating its heat, it's going back to a base state and a normal room temperature, eventually.

Joe Willmot (24:14):

On the left-hand side though, we have the situation where the heat generated is greater than the heat dissipated. Where we have an exponential release of energy at higher temperatures, the rates of reaction that we're observing within the cell are increased, that therefore releases more energy, that therefore heats up the cell even more, which then increases the kinetics, which then releases more energy. And that's where we get into a thermal runaway situation.

Joe Willmot (24:43):

Now we really want to be forcing ourselves into this bottom left-hand zone so that we know that if they do ever see these kind of situations, that if they fail or rather when they fail, they fail in as safe a manner as we can design them to. There are multiple ways for us to be able to achieve that. For example, the majority of cells these days have a pressure relief valve fitted in them or a pressure release device fitted within them. The reason that that's necessary is that it's obviously, if we're going to continue rising in heat, it's a much better idea to vent the excess pressure and in a safe manner, rather than waiting until it's at a point where the cell itself ruptures and starts spraying lithium all over the place. So you want to make sure that, all of those safety features that you've designed in the cell do work. And the way to do that is, the designs that you've taken to mitigate the cell failure do actually work. And the best way to facilitate this is with these extreme stress conditions.



Testing Procedures

Physical Damage
- Measure the effects of nail penetration

Overcharging and Discharge Rate Risks
- Discharging current, overcharging voltage that causes thermal runaway

Understanding the Effects of Battery Failure
- Amount of gas generated, visual battery damage

Temperature Effects
- At what temperature does battery start to self heat?
  - Define the maximum safe operating temperature
  - Main procedure is a Heat-Wait-Search test

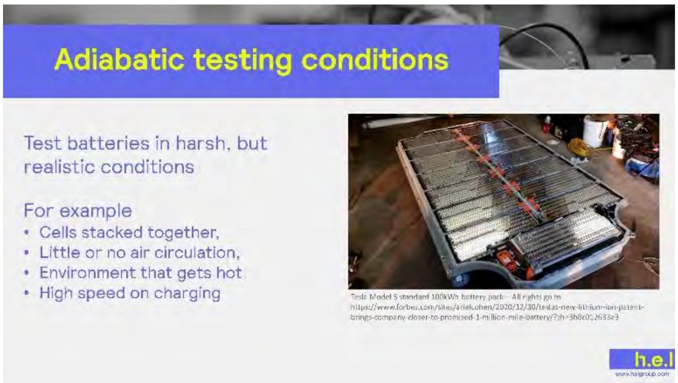
Making Batteries Safer
Better thermal management, information about internal events

Joe Willmot (25:53):

So some of the physical ones, as I mentioned, would be a nail penetration test. On the right-hand side, you see a cell before and after a nail penetration test. You can clearly see that, excuse me, you can clearly see that it can be very disastrous to mechanically stress the cell under operation. We've got the electrical abuse testing where we're deliberately overcharging and over discharging cells or batteries or entire battery packs if we're at this point of the assembly. And essentially all that this goes towards is understanding how the cell failed, why the cell failed and how could we mitigate that? How can we improve the cell design to ensure that that failure doesn't happen again? We've then also got the temperature effects to understand what temperature does the battery start to self-heat? Which will obviously define us the maximum safe working temperature of the battery or cell. The main procedure to do that would be a heat rate search test in an adiabatic calorimeter, which I'll just show you in a second.

Joe Willmot (27:01):

Essentially, as I said though, this is all for making batteries safe to potentially provide better thermal management or better information about what thermal failings are happening within the cell, to understand what can be done to improve them.



Adiabatic testing conditions

Test batteries in harsh, but realistic conditions

For example
- Cells stacked together,
- Little or no air circulation,
- Environment that gets hot
- High speed on charging

Tesla Model S standard 100kWh battery pack – All rights go to https://www.forbes.com/sites/arielcohen/2020/12/30/teslas-new-lithium-ion-patent-brings-company-closer-to-promised-1-million-mile-battery/?sh=3b8c012633e9

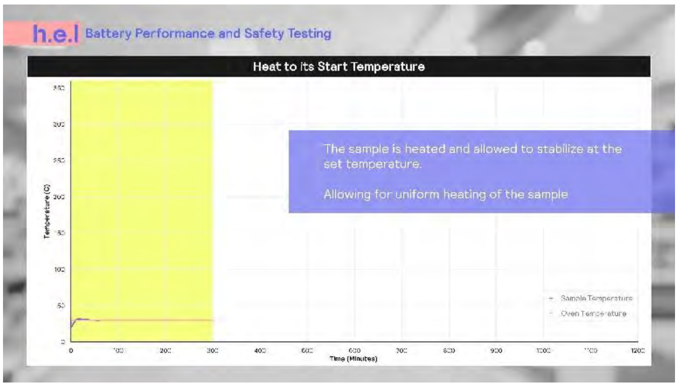
So as I mentioned, adiabatic conditions, why do we want to use adiabatic conditions? Well, adiabatic testing conditions, are essentially a representation of the absolute worst case scenario that a cell or battery or battery pack could find themselves in. Adiabatic conditions are when there are no heat losses from the cell, any energy that the cell generates are reabsorbed by the cell, go back into the cell to then again, increases rate of reaction, and potentially force it or take it to a point of thermal runaway. This is done by eliminating its heat losses, and that's where adiabatic calorimeters come in. Those are the testing pieces of equipment that allow you to eliminate the heat losses of your sample, so that when you're operating it, you're operating in an absolute worst case scenario.

Joe Willmot (28:15):

Now, it's not beyond the realms of possibility for cells to actually be used in adiabatic conditions. Electric vehicles are essentially the perfect storm for cells. If let's say the thermal temperature management system failed, well then as you can see with the picture of the Tesla battery pack, I've got a lot of cells in one place. I've got an extremely high capacity, I've got lots of energy, I've got high demands of extremely high charging and discharging rates. If I had a cell right there in the middle, I've got very little to no thermal cooling and no way to remove the heat from the sample. So if it goes into thermal runaway, I need to understand what that particular cell is going to do and how it's going to fail and ensure that it fails in a safe manner.

Joe Willmot (29:12):

You can take it one step further to even start testing full battery packs, meaning that you would take a whole pack of one of these batteries, and cause one of those cells to fail to see if there's thermal propagation, to see if one of the cell fails, does that affect the pack? Does that force the entire pack into a thermal runaway? Or have I designed enough of a protection to the rest of the cell so that I can draw power from all of them, but that they weren't thermally affect each other? So all of these considerations need to be apparent for electrical vehicle manufacturing, where adiabatic testing conditions aren't in a million miles away from what could actually be observed in individual cells.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Heat to its Start Temperature

The sample is heated and allowed to stabilize at the set temperature.

Allowing for uniform heating of the sample

Joe Willmot (29:59):

So to do the adiabatic testing, the most common way to provide this testing is with the heat rate search test. And to go through how this testing is carried out, you will place your sample into your adiabatic calorimeter and heat up to your start temperature. This allows any thermal difference in your sample, in your battery, in your cell, in your battery, in your battery pack, you want it all homogeneous. You want it all at the same temperature. You want it all in the same state and at the same temperature.


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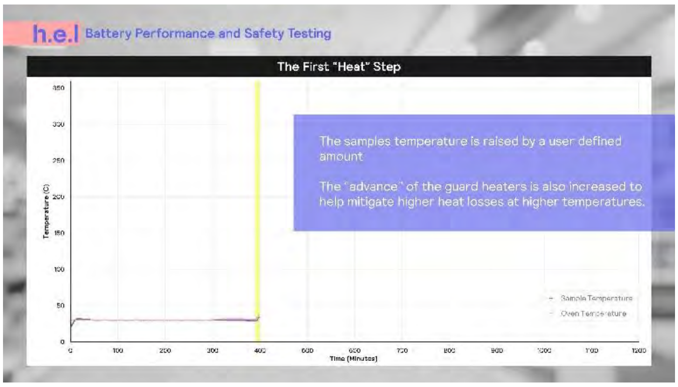
Online Calibration

The sample is being held under adiabatic conditions.

An "advance" is applied to the guard heater's temperature to mitigate heat loss.

We then adjust to allow for any slight variance between samples. Some cells are going to be slightly more energy dense than others, that will affect their thermal properties, and we have an adjust step to ensure that the guard heaters, the oven temperature increases to mitigate the sample's heat losses, so that any energy that the sample generates is retained within the sample.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The First "Heat" Step

The samples temperature is raised by a user defined amount

The "advance" of the guard heaters is also increased to help mitigate higher heat losses at higher temperatures.

Joe Willmot (31:06):

We then will increase in temperature, by use of defined mount, which is the first key step, so this is the heat part of the heat rate search. And the guard heat is advanced by a pre-calibrated amount to ensure that again, the heat losses are being mitigated.

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The First "Wait" Step

The sample is held adiabatically and allowed to settle at the elevated temperature.

The sample is then held adiabatically and allowed to settle at its elevated temperature, which is where we have the weight section of the fan name. There we allow it to all stabilize again, get all into the same phase.

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The First "Search" Step

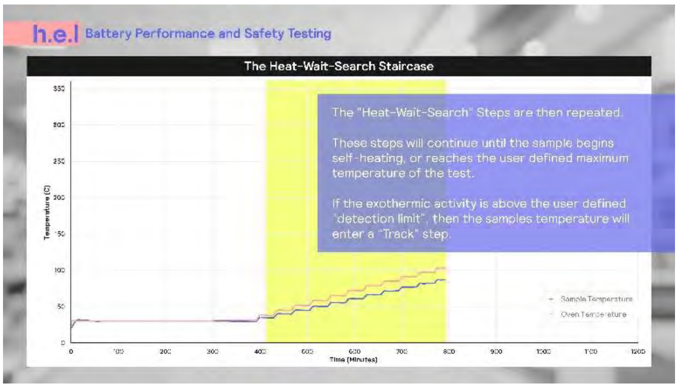
The samples temperature is monitored for any exothermic activity, known as the "Search" step.

If no exothermic activity is detected, then the samples temperature is raised by the user defined amount.

And then we finally have a search step. This is when the adiabatic calorimeter starts monitoring the sample temperature itself to see whether it's undergoing any exothermic activity. If the exothermic activity is above the user defined limit, then the adiabatic calorimeter will track that increase in sample temperature and increase the oven temperature, to again, eliminate any increased heat losses at the increased temperature that the sample is now at.

Joe Willmot (32:16):

Because obviously if I didn't do that, and the oven temperature remained constant, well then it's possible that the sample temperature then stabilizes at that higher temperature, but actually is losing heat to the calorimeter because I haven't compensated for the increased heat loss at the increased temperature. So what happens though for, hopefully down at these lower temperatures, is that that doesn't happen.



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The Heat-Wait-Search Staircase

The "Heat-Wait-Search" Steps are then repeated.

These steps will continue until the sample begins self-heating, or reaches the user defined maximum temperature of the test.

If the exothermic activity is above the user defined "detection limit", then the samples temperature will enter a "Track" step.

I have to repeat multiple of these heat rate search steps to get it to a point where I'm forcing the cell to start to fail. Now, there could be multiple reasons why the cell starts to fail. This particular point of cell failure could be the puncture point that you decide to pierce your cell at. It could be that this is the temperature, the separator between your anode and cathode starts to fail, starts to melt. If that is the case, then we know of cell manufacturers who have started making separators that actually block the transfer of lithium-irons, and therefore eliminate the possibility of a secondary thermal runaway.

Joe Willmot (33:37):

But in this case, it's quite possible that if that doesn't happen, that was a safety feature that they've designed in, they need to test to ensure that it works, that when the separator melts, it actually eliminates any potential of any additional thermal runaways. But if let's say they didn't have that, the normal separator melts causes one exotherm, does that then actually increase the cell's temperature to a point where it triggers a secondary exotherm? And that is actually what we see in this particular example.

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Self-Heating, Triggering the "Track" Phase

The sample begins self-heating and adiabatic conditions are maintained. Any release of thermal energy that is retained within the sample, further increasing its temperature.

The system remains in the "Track" step until the sample reaches a user defined limit or the exothermic event has finished, and the test shuts down.

Joe Willmot (34:12):

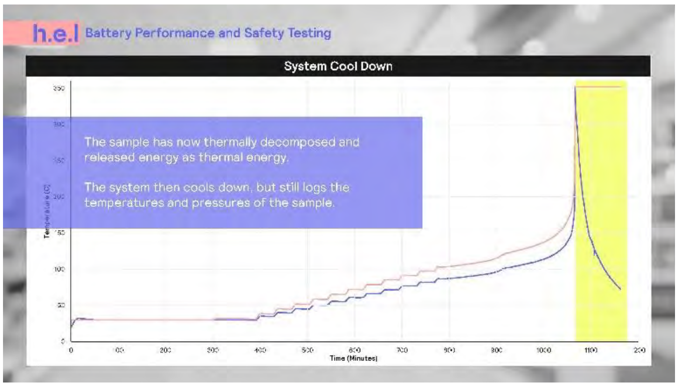
The sample begins self-heating, adiabatic conditions are maintained. We then track the sample temperature and the system remains in track until the sample temperature reaches a user defined limit, or the temperature stabilizes out. If let's say we have an initial exotherm that then stabilizes out, we would then go back into heat rate search that's until we've reached the user defined limit of what the maximum temperature is.

Joe Willmot (34:41):

So for this particular example, this was an 18, 650 test cell for this graph here. So you can see that we've got the sample temperature in blue, starting to run away. It hits this into its own where obviously there's then another internal change inside the cell, and then we hit a secondary exotherm that then shoots up right, the way up to over 350 degrees C, which is what the maximum temperature for tracking the exotherm was in this particular test.

Joe Willmot (35:15):

You can see that the rate of these exotherms is something to be reckoned with. You can easily get two realms of 300, 400, 500 degrees C per minute if not more, maybe even in the thousands, depending on how energy dense your sample is. And at that point the sample becomes pseudo adiabatic, which means that the sample cannot lose its heat any quicker than it's already increasing. So it doesn't have any time to lose any of its heat, all this remains within the cell so that the adiabatic calorimeter is trying to keep up, but it's going at such a faster rate that essentially it's pseudo adiabatic, all the energy is retained within the sample because it hasn't had any time to dissipate any of that energy. And once we've reached that final temperature, we then go into a shutdown state for this particular adiabatic calorimeter, where we just stop heating and we pull the sample back down.



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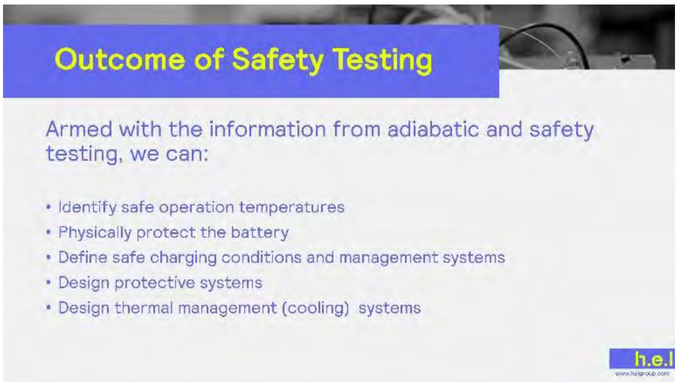
System Cool Down

The sample has now thermally decomposed and released energy as thermal energy.

The system then cools down, but still logs the temperatures and pressures of the sample.

Joe Willmot (36:20):

So you can see how running these adiabatic tests allow us to test these samples in absolute worst case scenarios. And we can start to characterize, if we look at their thermal runaways and look at what happens to them, we can start to characterize certain internal cell failings that might lead to additional cell failings. We can also see, if we make a change to the cell's design, if we change the electrolyte, that then has a much lower vapor pressure, that then doesn't produce as much toxic gases from the cell. Like that's definitely an improvement in safety, that will need to be tested to be determined and confirmed.



Outcome of Safety Testing

Armed with the information from adiabatic and safety testing, we can:

- Identify safe operation temperatures
- Physically protect the battery
- Define safe charging conditions and management systems
- Design protective systems
- Design thermal management (cooling) systems

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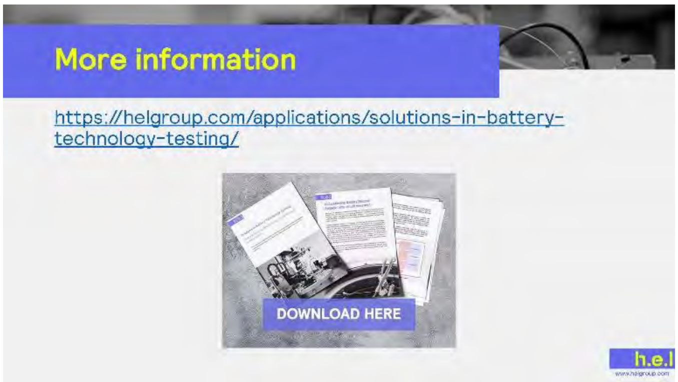
Joe Willmot (37:07):

So really all of this, what's the outcome of the safety testing? Under the information of adiabatic calorimetry, we can identify the maximum safe temperature of operation, at which point does my first thermal runaway start to happen? And therefore that is my maximum temperature of use. I will not ever use a battery above that temperature. Physical protection with my nail penetration tests, have I got sufficient physical protection to the battery so that if it's exerted under X amount of force, it will still be okay? We can define the self-charging and discharging of the cell via us maintaining it under adiabatic conditions and just operating it normally, and seeing whether that actually takes it into a thermal runaway, and if it does, we need to ensure that the thermal management system can adequately ensure that it doesn't ever go above those temperatures. Is there a block that we could design that melts to ensure that the battery is always having its heat dissipated effectively from it, et cetera, et cetera.



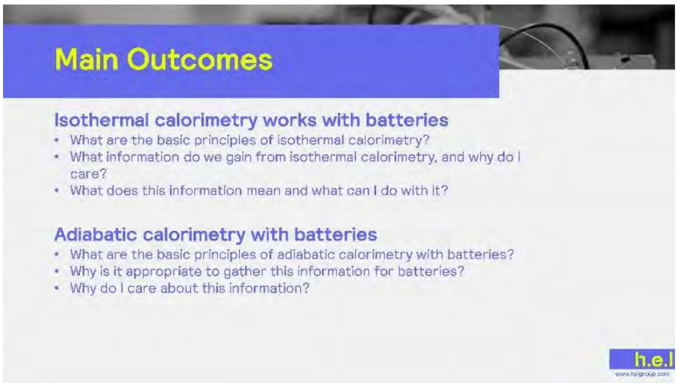
More information

https://helgroup.com/applications/solutions-in-battery-technology-testing/

Read Now: [Solutions in Battery Technology Testing: Hazard screening, safety testing and performance characterization]()

Joe Willmot (38:20):

All of that information comes out of adiabatic testing and helps with better cell design. The more information around anything that I've discussed today, we do have our, understanding battery thermal behavior brochure and a little leaflet for you to go and read, where we cover a whole wide range of why the safety testing needs to run in parallel to the performance testing, where the compromises might need to be made and what the risks are associated to them. So please do go away to that website, have a read and let us know your thoughts about it.



Main Outcomes

Isothermal calorimetry works with batteries
- What are the basic principles of isothermal calorimetry?
- What information do we gain from isothermal calorimetry, and why do I care?
- What does this information mean and what can I do with it?

Adiabatic calorimetry with batteries
- What are the basic principles of adiabatic calorimetry with batteries?
- Why is it appropriate to gather this information for batteries?
- Why do I care about this information?

Joe Willmot (39:03):

So in summary, the main outcomes from today, we wanted to see what performance testing was, what isothermal calorimetry was. We understand what the basic principles are, that we won't actually eliminate the heat losses, mitigate, sorry, not eliminate, mitigate the heat losses by holding the sample isothermally, to be able to measure the thermal duty under normal operation. What information do we gain from isothermal calorimetry? We've seen that we gain information about the thermal duty that the cell is undergoing, and how that could potentially help us design a thermal management system to the cell or battery pack to ensure that we have sufficient cooling present at all times, to ensure that the temperature doesn't go above a particular efficiency temperature that we get the most efficiency from the cell.

Joe Willmot (39:58):

For adiabatic testing, obviously that information that we have from the isothermal feeds into the safety, sometimes where we have, under normal operation we release X amount of heat, if we release X amount of heat, what will that do to my final temperature? Is that final temperature actually going to take it above my thermal onset temperature of thermal runaway that I've determined from adiabatic calorimeter? All the other information are going to be joined together from isothermal and adiabatic testing. So that it covers... Oh, the basic principles also is obviously to eliminate your heat losses and test the battery under an absolute worst case condition, that hopefully it will never see in the real world, but it's not beyond the realms of possibility for it to operate under. And if it does operate under those conditions, that if it does fail, it fails in as safe a manner as we can. We could reduce the final temperature that it gets to, by potentially changing some internal components. We could ensure that it releases as little toxic gases as possible and go from there.

Joe Willmot (41:17):

And as I said, this is how we envisioned both isothermal calorimetry and adiabatic calorimetry coming together, to provide us with a more comprehensive picture of how changing certain aspects of the cell can help improve on cell design, eventually leading to safer and better batteries.

Thank you for listening

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Joe Willmot (41:38):

So thank you all very much for listening to me today. I would like to thank, obviously the opportunity to talk here today. It was very gracious to be allowed to talk. If you have any information, if you have any questions, sorry, please do check out our website, or if you wish you can contact me directly. My email's on the screen now and I'll happily take any questions that may have come up in the chat.

Shlomit Dery (42:04):

Okay. So we have one request for the presentation to be be forwarded. Is that possible?

Joe Willmot (42:14):

Yes, yes, that should be possible. I'll happily send these slides over.

Shlomit Dery (42:16):

Okay. So I will send it to you guys. Thank you very much, Joseph. And thank you for all of you customers, attendees that joining us. And I remind you that there will be now will be upload in the following days to the website, and you can share it if you want.

Joe Willmot (42:37):

Yes, definitely.

Shlomit Dery (42:41):

And if you want to go deeper, you can always call Emmanuel or me, and we will fit our solution to your challenges. Of course, with Joseph in the background, pushing us to the solutions for you. Don't forget to follow us in LinkedIn for more data and updates. Happy holidays guys. Thank you.

Joe Willmot (43:02):

Thank you very much.