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Electrochemical Performance of Lac Knife Natural Crystalline Flake Graphite from Quebec, Canada in Lithium Ion Batteries

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Summary

Initial galvanostatic electrochemical testing of coin cells showed that Lac Knife spherical flake graphites (SPGs) achieved nearly theoretical reversible capacities (RCs) and low irreversible capacity losses (ICLs) of 1% compared with relatively low RCs of 345 and 347 mAh/g and high ICLs of 3.8 % and 6.5% obtained with commercial synthetic graphites. Long term cycling tests conducted on Lac Knife SPG exhibited zero RC loss after 200 cycles and only a 4.5% loss when the tests ended after 560 cycles which compares with losses of over 10.5% reported on commercial SPGs after 420 and 510 cycles.

Tests also were conducted on Lac Knife SPG containing 4.5% and 18% amorphous silicon which resulted in an increase of the RC of the silicon enhanced SPG to 462 and 613 mAh/g, well beyond the theoretical limit of 372 mAh/g for graphite.

Keywords: spherical graphite (SPG), reversible capacity, irreversible capacity loss (ICL), long term cycling, silicon

1 Lac Knife Graphite

1.1 Location of Lac Knife Graphite Deposit

The Lac Knife graphite deposit is located near the Labrador border, south of Fermont, Quebec. It contains measured and indicated reserves of approximately 12.6 million tons of 15% graphitic carbon, making it one of the highest-grade graphite resources in the world. The area hosts excellent infrastructure with low cost Quebec hydroelectric grid power and nearby access to road and rail with access to port facilities along the St. Lawrence Seaway. The deposit is also close to Wabush and Labrador City which serve as bases for three iron ore mines in the area, one being the largest open pit mine in North America.

1.2 Upgrading of Lac Knife Graphite Ore

Extensive pilot plant flotation tests were conducted in 2013 at SGS Laboratories Inc. on 23 tons of crushed and sized ore recovered from representative cores collected from the drill program ore taken from the massive, semi-massive and low grade mineralization zones of the Lac Knife deposit. The average grade of the coarse, +80 mesh fraction recovered in the flotation tests was 98.3% total carbon ("Ct") compared with 98.2% Ct and 98 % Ct for the medium, -80 x +150 mesh and fine, -150 x + 200 mesh fractions recovered from the flotation

tests. The average carbon content of all size fractions for the pilot campaign was 96.6% C. It is important to note that these results were achieved despite the fact that the -200 mesh fines fraction was not subjected to a fines cleaning circuit which would be installed in a full scale plant.

These results indicate that the impurities present in the Lac Knife graphite are attached to the surface of the flakes in the flotation concentrate and, hence, can be removed by standard upgrading processes. This is shown in Fig. 1 where scanning electron microscope (SEM) photomicrographs of the flotation concentrate, purified flake and spherical graphite are compared.

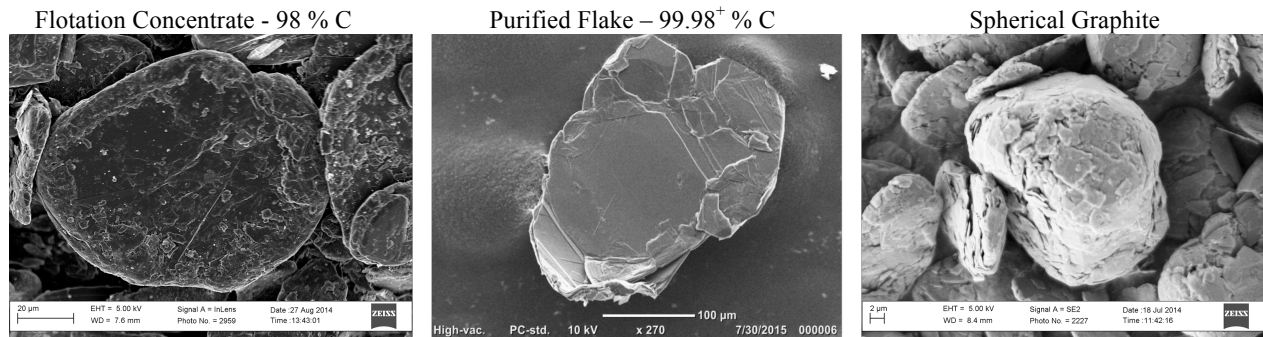


Figure 1: SEMs of Upgraded of Lac Knife Graphite

The SEM of the flotation concentrate shows that the impurities are present on the surface of the individual flakes as white specks. More detailed information on the Lac Knife graphite deposit, drilling program and the upgrading of the graphite from the drill core samples can be found in the presentation made at the 32nd International Battery Seminar and Exhibit on March 10, 2015^{1,5}.

1.3 Current Status of the Lac Knife Graphite Mine Development Project

In 2014 Focus Graphite announced the completion of a Feasibility Study (“FS”) for the Lac Knife project conducted by Met-Chem Canada Inc. based on the production of 44,300 MT of flake graphite annually over a 25 year mine life with an average carbon content of 97.8 wt % C. The FS was based on the pilot plant test work described in Section 1.2 above. AGP Mining Consultants Inc. prepared the National Instrument Mineral Resource Estimate (“NI 43-101”) for the project which estimated a total of 9.6 million tons of measured and indicated reserves at a carbon content of 14.8 wt% C.. In 2017 AGP updated their mineral reserve estimate resulting in an increase in the measured and indicated reserves by 42% to 14.6 million tons at a carbon content of 15.0 wt% C. Work is continuing with regard to obtaining environmental permitting and the submittal of mine closure plans for the Lac Knife project.

2 Electrochemical Performance of Lac Knife Coated Spherical Graphite

2.1 Purification of Lac Knife Graphite

The upgraded, 98.3 wt% C graphite described in Section 1.2 was dried, screened and thermally purified to achieve the desired purity level for electrochemical power sources. This is important because the presence of many impurities in graphite have been shown to be detrimental to battery systems as they may be the source of in-cell gassing and/or increased irreversible capacity loss. The SEM of the Lac Knife flake graphite after thermal purification in Figure 1 shows that essentially all of the impurities have been removed from the graphite flakes which increased the carbon content of the purified flake to 99.98⁺ wt% C and is well above the recognized minimum purity level of 99.95 wt% C required for electrochemical power sources.

2.2 Preparation of Carbon Coated Lac Knife Spherical Graphite

The purified flake from Section 2.1 was mechanically spheroidized in a proprietary process in such a way as to both reduce the size of the graphite flakes while folding individual flakes of graphite into small spheres. This is shown in the SEM of spherical graphite in Figure 1 where the flakes of graphite are clearly folded on top of each other to form a spherical graphite particle.

The reason why spherical particles are needed is because in a scenario of random packing (which is the case of a typical lithium ion anode coating), spheres offer maximum density among all other shapes. The spherical graphite was then coated with an ultra-thin layer of amorphous carbon using a proprietary coating process to further increase the packing density of the anode matrix which will maximize both the Specific Energy (measured in Wh/kg) and Energy Density (measured in Wh/L) of a full battery.

The exterior carbon shell must be as thin as possible (e.g. nanosized), and completely permeable to the ingress and egress of Li^+ ions into the graphitic core. The carbon coating serves three important functions: it prevents the start of a thermal runaway by reducing the BET surface area of graphite; it reduces dramatically the irreversible capacity loss on the anode; and it functions as a cushioning substrate onto which the deposition of polymer binder will subsequently occur.

2.3 Development of Standard, Fine and Superfine Grades of Coated Spherical Graphite

Both the standard ($D_{50} = 23.9 \mu\text{m}$) and fine ($D_{50} = 17.4 \mu\text{m}$) grades of uncoated and carbon coated Lac Knife spherical graphite (SPG) were developed and tested in 2014 and reported on at the presentation made at the 32nd International Battery Seminar and Exhibit on March 10, 2015^{1,5}. The particle size distributions were determined by laser diffraction using a Microtrac S3500 particle size analyzer and the grades developed were tested for their main physicochemical and electrochemical properties to evaluate their performance as an active material in the negative electrodes of lithium ion batteries. The superfine ($D_{50} = 11.9 \mu\text{m}$) grade of carbon coated SPG was developed in early 2017 in order to serve the increasing demand for a finer sized grade of SPG in high energy power applications. The particle size distribution curves of the three grades of SPG are shown in Figure 2.

Included in the figure are the ranges in D_{50} s for each of the three grades with the new superfine grade having a D_{50} in the range of 10 to 15 μm .

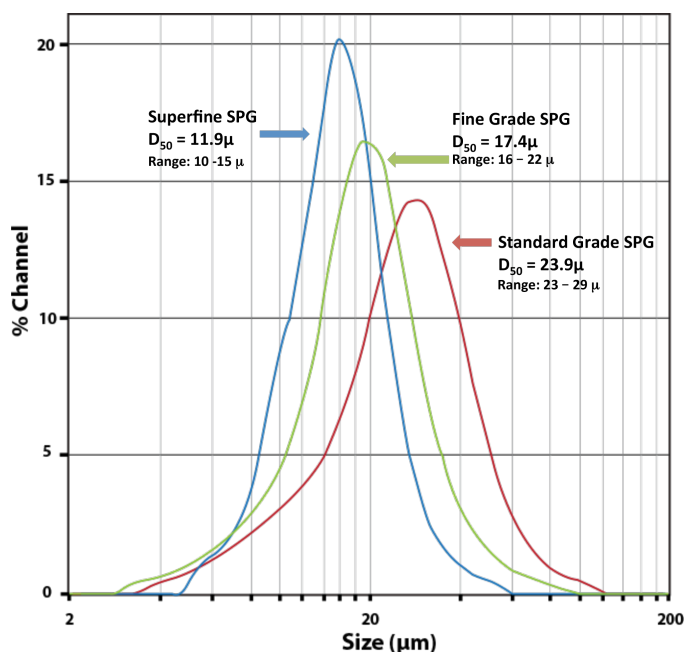


Figure 2: Particle Size Distributions of Superfine, Fine and Standard Grades of Lac Knife SPG

2.4 Electrochemical Performance of Superfine Grade of Coated Spherical Graphite

The initial electrochemical performance of both the new superfine grade of coated spherical graphite and the standard and fine grades were determined using standard CR2016 coin cells. A cross sectional schematic of the actual CR2016 coin cells used can be found in the presentation made at the 33rd International Battery Seminar and Exhibit on March 21, 2016^{2,5}. Although a number of performance parameters have been studied, this section specifically focuses on the performance of the cells during their formation cycle. Specifically, in the paragraphs which follow, the values of reversible and irreversible capacities at a C/20 cycling rate in half cells and the irreversible capacity loss on the formation vs Li/Li^+ counter electrode in the CR2016 coin cell are reported for the three grades tested. The electrolyte used was 1.0M LiPF_6 in FEC/EMC (30:70 vol) with an 8 mil thick electrode whose composition is 90 wt% graphite and 10 wt% supporting additives, including a PVDF based binder and a VC additive.

The initial galvanostatic charge-discharge curves for the new superfine grade of Lac Knife coated SPG are given in Figure 3.

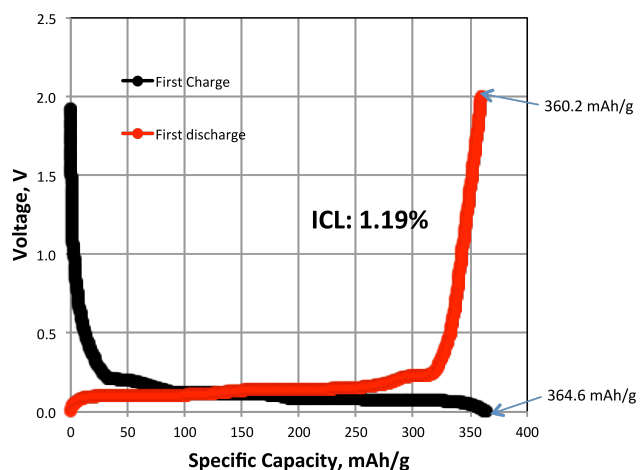


Figure 3: Initial Galvanostatic Charge-Discharge Curves for Superfine Grade of Coated SPG

As shown the reversible capacity of the new superfine grade at 360 mAh/g compares very favorably with the reversible capacities of the standard and fine grades of the Lac Knife coated SPG at 364 and 365 mAh/g, and all are close to the theoretical capacity of 372 mAh/g for graphite^{3,5}. Likewise the low Irreversible Capacity Loss of 1.19% for the new superfine grade of SPG also compares very favorably with the ICLs of the standard and fine grades at 0.48 and 1.44%.

Surface areas were determined using a Quantachrome Instruments Monosorb™ BET Surface Area Analyzer. The surface area of 0.89 m²/g shown in Table 1 for the new superfine grade of SPG is almost in the middle of the surface areas of 0.48 and 1.14 m²/g for the standard and fine grades. What is important here is that all three grades are very low in surface area which is good for preventing thermal runaways in lithium-ion batteries. These results also show the importance of coating the SPG with carbon because the surface area of the uncoated standard grade of SPG is 5.15 m²/g compared to the coated version at 0.48 m²/g.²

As shown, the irreversible capacity for the first charge is 364.6 mAh/g and the reversible capacity for the first discharge is 360.2 mAh/g thereby obtaining an Irreversible Capacity Loss (ICL) of 1.19%. The original Galvanostatic curves for the standard and fine grades of SPG are available in the presentation made at the 32nd International Battery Seminar and Exhibit on March 10, 2015^{1,5}.

A comparison of the electrochemical performance characteristics and the surface areas of the standard, fine and superfine grades of carbon coated SPG is provided in Table 1.

Table 1: Comparison of the Electrochemical Performance of Lac Knife Spherical Graphites

	Reversible Capacity (mAh/g)	Irreversible Capacity Loss %	Surface Area (m ² /g)
Standard Carbon Coated SPG Grade (D ₅₀ = 23.9μ)	363.7	1.44%	0.48
Fine Carbon Coated SPG Grade (D ₅₀ = 17.4μ)	365.1	1.01%	1.14
Super Fine Carbon Coated SPG Grade (D ₅₀ = 11.9μ)	360.2	1.19%	0.89

3 Comparison of the Electrochemical Performance of Lac Knife Spherical Graphite with Commercial Grades of Synthetic Graphite

3.1 Grades of Lac Knife Coated Spherical Graphite and Synthetic Graphite Tested

The electrochemical performance of the fine (D₅₀ = 17.4 μm) grade of Lac Knife carbon coated SPG described in Section 2 was compared with the electrochemical performance of two commercial grades of Synthetic Graphite. The two commercial grades of synthetic graphites were Xiamen TSAG-H (identified as Synthetic Graphite #1) and Xiamen TS360 (Identified as Synthetic Graphite # 2). Both of the Xiamen grades had a D₅₀ of approximately 20 μm. The same procedures described in Section 2 to prepare and test the coin cells used with the Lac Knife carbon coated SPG were used to prepare and test the coin cells with synthetic graphite.

3.2 Comparison of Initial Galvanostatic Charge-Discharge Curves for Lac Knife Spherical Graphite and Synthetic Graphite

The initial charge-discharge curves for the fine grade of the Lac Knife carbon coated SPG are compared with the two grades of synthetic in Figure 4.

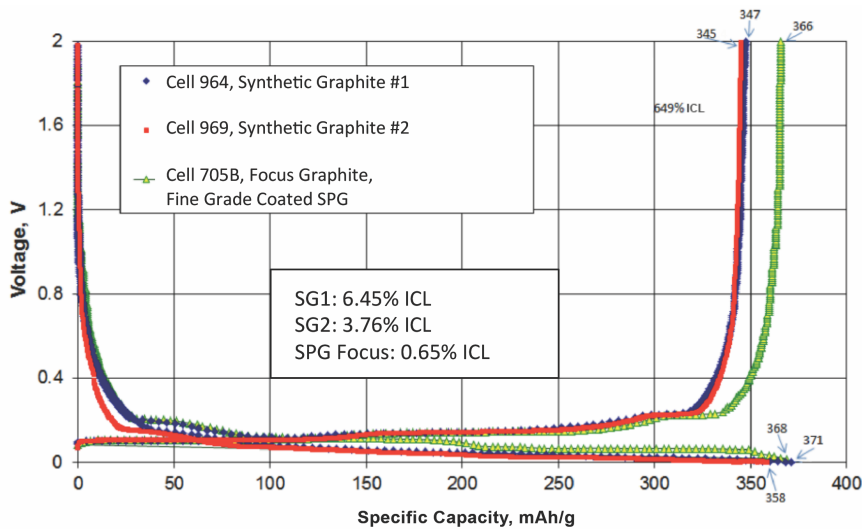


Figure 4: Initial Charge-Discharge Curves for Lac Knife Flake Graphite Compared with Synthetic Graphite

As shown the two grades of synthetic graphite tested had reversible capacities of 345 and 347 mAh/g or about 5% below the reversible capacity of 366 mAh/g achieved with the Lac Knife SPG. Furthermore the Irreversible Capacity Losses (ICLs) of the Synthetic Graphites #1 and #2 were 6.45% and 3.76%, much higher than the ICL of 0.65% achieved with the fine grade of Lac Knife SPG.

3.3 Galvanostatic Curves for Lac Knife Spherical Graphite at Varying Charge Rates

The fine grade of Lac Knife carbon coated SPG was cycled in coin cells at using the following cycling protocol: 3 cycles at C/20, 2 cycles at C/10, 1 cycle at C/5 and 20 cycles at C/2. Only the cycling results for the C/20, C/5 and C/2 rates are presented in Figure 5.

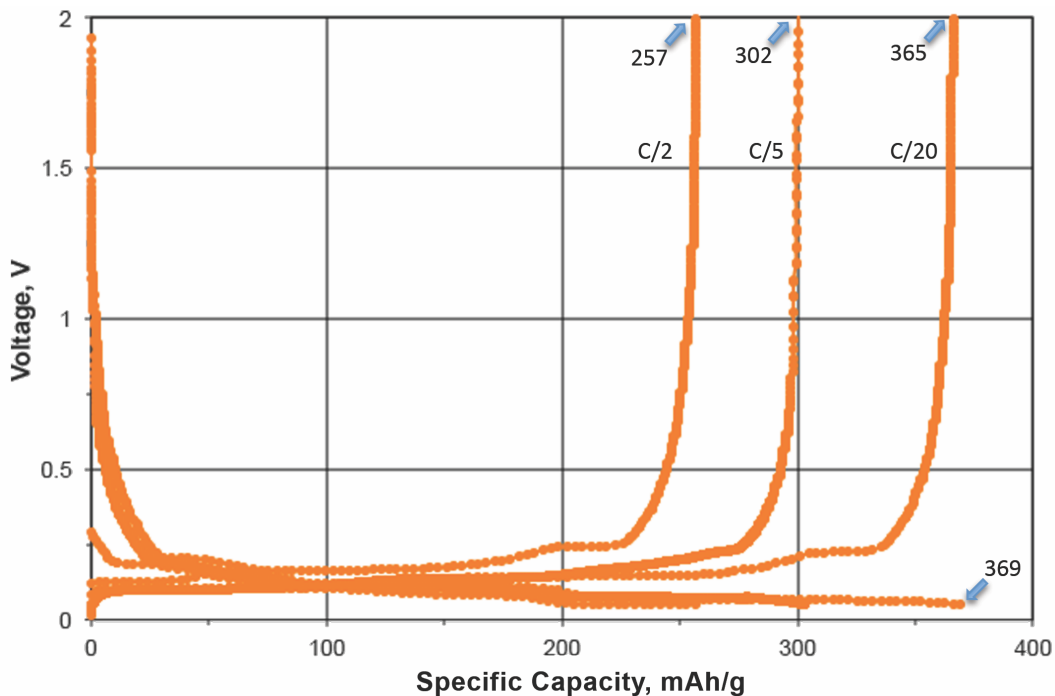


Figure 5: Galvanostatic Charge-Discharge Curves for Fine Grade of Carbon Coated SPG At C/20, C/5 and C/2 rates in CR2016 Half Cells.

The curves shown in Figure 5 show the results for the fine grade of SPG as it cycles three times through a C/20 charging rate at a reversible capacity of 365 mAh/g and followed by the C/5 and C/2 charging rates. It should be pointed out that this cycling protocol is not a standard testing protocol because of the design limitations of the coin cells. However, of practical value is the result demonstrated at the C/2 rate which refers to a two-hour long charge and two-hour long discharge which is a profile similar to the active use of a cellular phone. A surprisingly high reversible capacity of 257 mAh/g was achieved with the fine grade of coated SPG at the C/2 rate without any optimizations done to the system.

3.4 Comparison of Galvanostatic Curves for Lac Knife Spherical Graphite and Synthetic Graphite at Varying Charge Rates

The coin cells made with synthetic graphite were cycled at varying charging rates using the same cycling protocol used for the Lac Knife fine grade of coated SPG in Section 3.3. In this case, however, only the cycling results comparing the C/20 and C/2 charging rates for both the Lac Knife and synthetic graphites are shown in Figure 6.

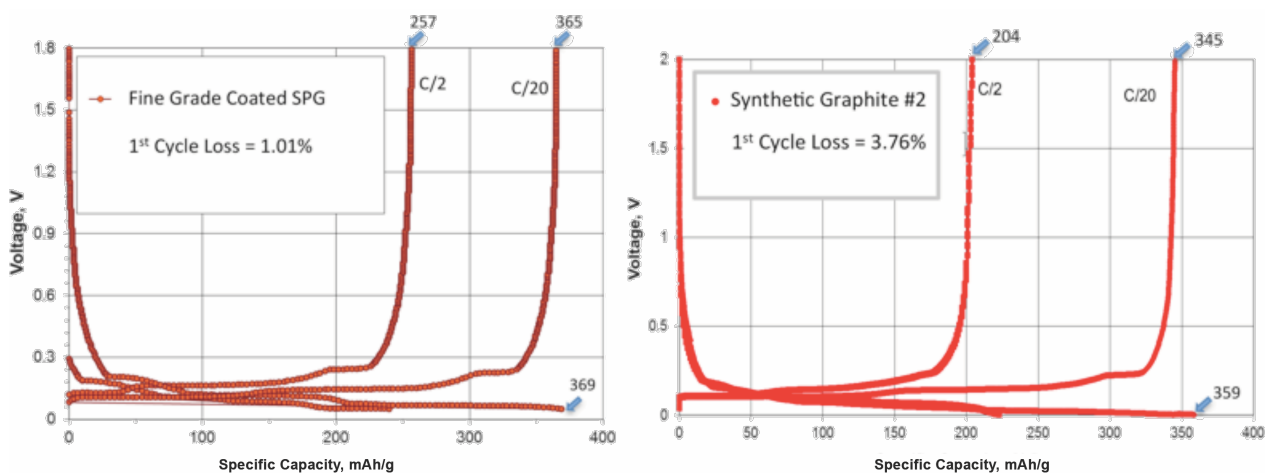


Figure 6: Charge-Discharge Curves for Carbon Coated SPG & Synthetic Graphite at C/20 and C/2 Rates in CR2016 Half Cells

Although it was pointed out that the varying cycling protocol used in Section 3.4 is not a standard protocol to use in coin cells, it can be used to compare the cycling performance of two different graphites as long as they are tested using the same protocol. In this case the difference in the reversible capacity of 365 mAh/g in the coin cell made with Lac Knife SPG and the coin cell made with synthetic graphite of 345 mAh/g at the C/20 charging rate is even more pronounced at the C/2 rate where the reversible capacity of the Lac Knife coin cell is 257 mAh/g compared with 204 mAh/g for the synthetic graphite coin cell.

4 Comparison of the Long Term Cycling Performance of Lac Knife Flake Graphite with Commercial Grades of Natural Flake Graphite

4.1 Preparation and Testing of Anodes for Long Term Cycling Tests

The anodes prepared for the long term cycling tests consisted of Lac Knife fine grade of carbon coated spherical graphite (90%), PVDF binder (7%), Imerys C-ENERGY™ SUPER C65 Carbon Black Additive (3%) and Cu foil current collector (thickness 20µm). The Lac Knife coated SPG had a D_{50} of 18.94 µm and a BET Surface Area of 0.82 m²/g. The slurry for electrode casting was prepared from a mixture of the Lac Knife graphite and Solvay's Solef 5130 PVDF binder dissolved in 1-methyl-2- pyrrolidinone (NMP). The slurry was spread onto a Cu foil and dried under vacuum at 120° C for 12 hr. After drying, the electrodes were compressed by roll press for achieving electrode density 1.2 g/cm³.

All cells were assembled for testing in an Argon-filled glove box. Anodes were tested in coin cell (CR2016) configurations prepared with 1M LiPF₆/EC/DMC electrolyte and Li foil reference/counter electrodes. The coin cells were cycled between 0.003 and 1.5 volts. Formation was carried out with C/10 current density and cycling was carried out with the same voltage limits at C/10.

4.2 Comparison of the Long Term Cycling Performance of Lac Knife Coated Spherical Graphite with Commercial Grades of Coated Spherical Graphite

The long term cycling performance of the Lac Knife coated SPG was compared with Changsha Hairong's SKG-1 grade of coated natural flake spherical graphite (SPG) using available data. The SKG-1 coated SPG has a D₅₀ of 19.78 μm and a BET Surface Area of 1.56 m²/g. The results from the long term cycling tests comparing Lac Knife coated SPG with the SKG-1 (identified as Supplier Discharge Capacities #1 and #2) coated SPG are presented in Figure 7 below:

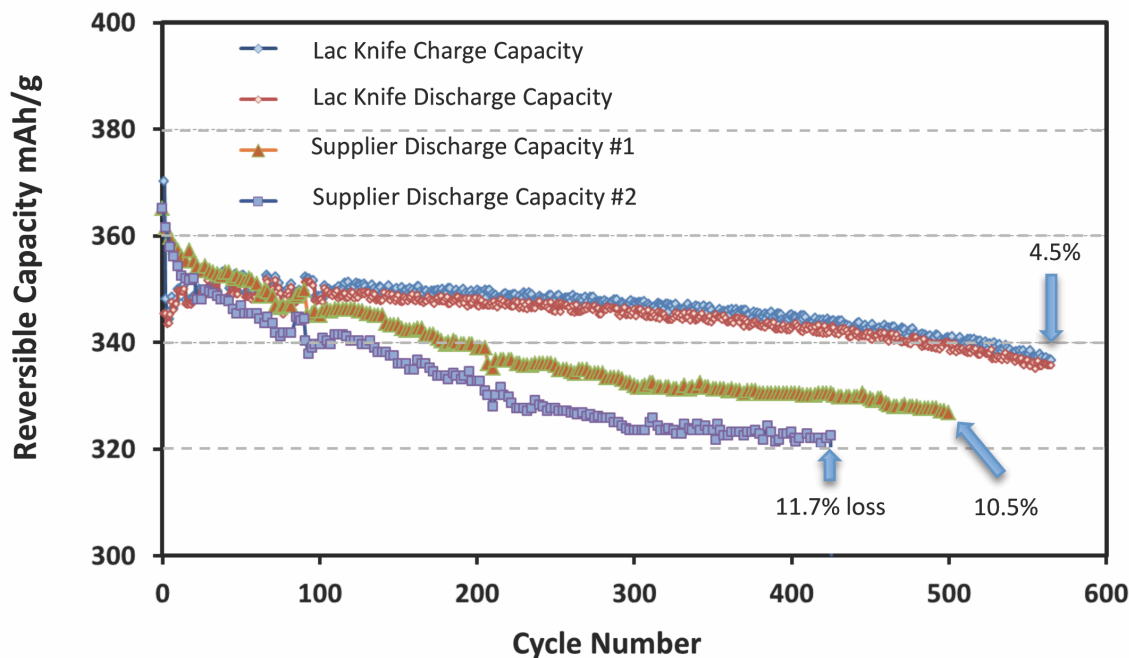


Figure 7: Long Term Cycling Performance of Lac Knife Graphite Compared with Two Commercial Grades of Coated Spherical Graphite

As shown, the long term cycling tests conducted on Lac Knife carbon coated SPG in the anode of CR2016 configured coin cells exhibited almost zero loss of capacity after about 200 cycles as compared with the two tests run on the commercial supplier's grade of carbon coated SPG which started to decline in capacity almost immediately after the first cycle. The long term cycling tests on the Lac Knife SPG continued until the tests finally ended at about 560 cycles with only a 4.5% capacity loss. This compares with capacity losses of 11.7% and 10.5% reported on the two tests run on the commercial supplier's grade of coated SPG at the conclusion of their tests at 420 and 510 cycles.

5 Comparison of the Conductivity of Lac Knife Expanded Flake Graphite with Commercial Grades of Graphite in Cathodes of Li Ion Batteries

5.1 Preparation of Expanded Graphite

Expanded graphite is a form of processed natural crystalline flake, featuring dramatically improved electrical conductivity in cathode mixes which is critical in the design of lithium ion batteries where much of the effort is concentrated on improving the performance of the anode. The expanded graphite used in this study was produced by first adding Lac Knife purified flake graphite into a blend of sulfuric and nitric acids at room temperature using a proprietary process to produce an intercalated flake graphite. The intercalated graphite was

then neutralized and dewatered in a Buechner funnel prior to introducing the graphite into a muffle furnace at 950°C to produce expanded “worms” of graphite having an expansion volume of 250⁺ mL/g and about an 18% weight loss. The expanded graphite worms were then air milled to delaminate and produce the desired particle size distribution. The specific details regarding the process used to produce expanded graphite can be found in the presentation made at the 33rd International Battery Seminar^{2,5} on March 21, 2016.

SEMs of a side view of a purified flake of graphite and the resultant expanded Lac Knife graphite are shown in Figure 8 below:

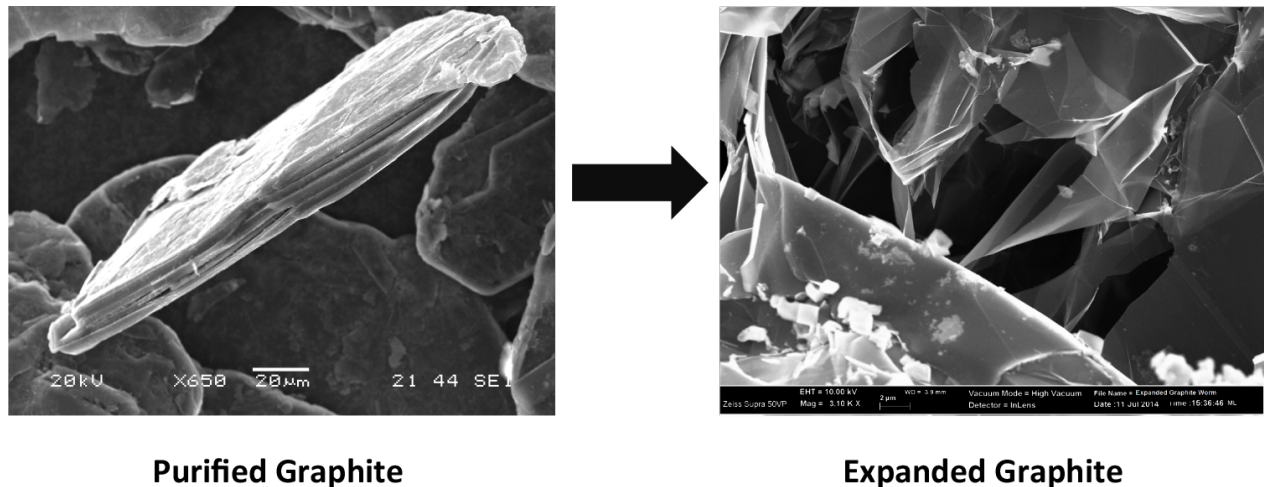


Figure 8: Scanning Electron Photomicrographs of Purified and Expanded Lac Knife Graphite

As can be seen in the SEM on the left, the graphite flakes consist of many layers of individual thin flakes of graphite which are intercalated with acids and then expand like an accordion when introduced into the furnace. The resultant expanded flakes shown on the right consist of many thin, almost translucent nano-sized flakes of graphite which are highly conductive when compared with the standard flake and synthetic graphites that are used in the cathode matrices of lithium ion batteries.

5.2 Development of Test Method to Compare Resistivities of Graphite Samples

A four point resistivity tester was designed and manufactured based on using a modification of the ASTM C 611 Test Method in order to be able to compare the conductivity of the Lac Knife expanded graphite with the standard flake and synthetic graphites used as conductive additives in the cathodes of lithium ion batteries. The four point resistivity tester was introduced along with schematic drawings at the 33rd International Battery Seminar^{2,5} on March 21, 2016 and is shown in Figure 9.

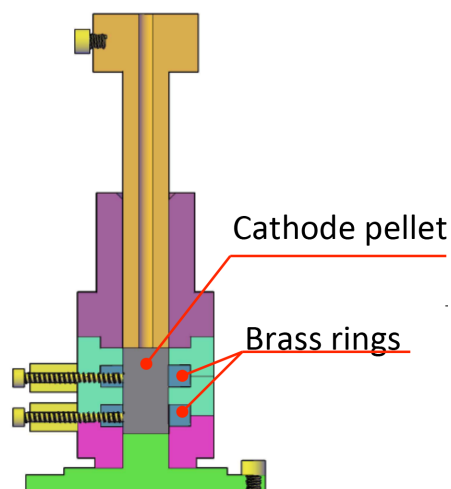


Figure 9: Cross Section of Four point Resistivity Tester

5.3 Comparison of the Resistivities of Lac Knife Expanded Graphite with Commercial Grades of Flake and Synthetic Graphites in Lithium Ion Batteries

It is customary to compare the conductivities of different materials used in the matrices of cathodes by plotting and comparing the resistivity of the cathode matrix as function of the percentage of the type of graphite added to the matrix. This is shown in Figure 10 where the resistivity of two grades of expanded Lac Knife graphite are compared with the resistivities of commercial grades of flake and synthetic graphites in a formulated cathode matrix where Lithium Nickel Manganese Cobalt Oxide (LiNiMnCoO_2) is the active material.

Figure 10 shows that replacing commercial grades of synthetic and flake graphites with the Lac Knife expanded graphite in the cathode of a lithium ion battery greatly reduces the resistivity of the cathode matrix thereby greatly increasing the conductivity. As shown, if a target of 1 ohm-inches is set for the resistivity of the cathode mix, the amount of expanded graphite that needs to be added to the mix is only 1% as compared with 2.5 to 3.5% for the commercially used standard synthetic and flake graphites. Conversely, at a concentration of 2 wt% graphite, the resistivity of the cathode matrix using either flake or synthetic graphite is in the range of 1.4 to 1.9 ohm-inches. Replacing the standard graphites currently being used as conductive additives in the cathode matrix with 2 wt% of the Lac Knife expanded graphite, reduces the resistivity of the matrix to a range of 0.2 to 0.35 ohm inches, resulting in an increase in the conductivity of the cathode matrix by a factor 4 to ten times depending on which grade of expanded graphite is used^{4,5}.

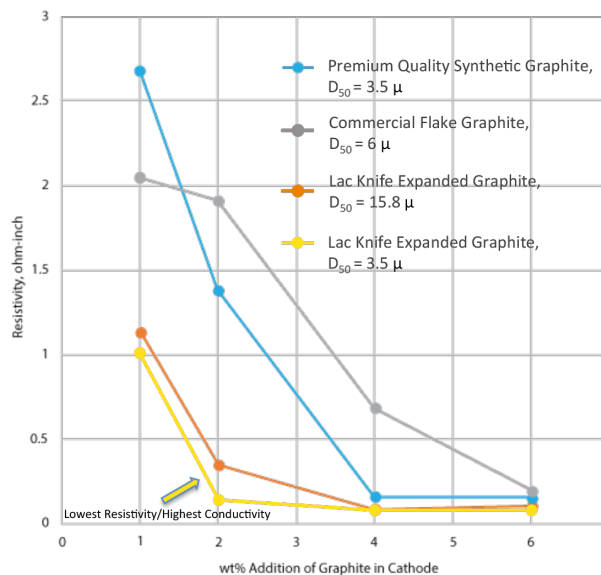


Figure 10: Comparison of Resistivities of Lac Knife and Commercial Graphites in Lithium Ion Cathode Matrix (LiNiMnCoO_2)

This is important because this gives the battery manufacturer greater flexibility in the formulation of the design of the cathode to take advantage of the continuing improvements being made in anode formulations. One such benefit would be to replace the standard graphite in the cathode matrix by adding a much smaller percentage of the Lac Knife expanded graphite thereby allowing for more of the active material to be included in the cathode mix thereby providing the opportunity to either increase the capacity or extend the life of the lithium ion battery.

6 Electrochemical Performance of Lac Knife Silicon Enhanced Spherical Graphite

6.1 Addition of Silicon to Increase the Reversible Capacity of Flake Graphite

In scientific terms, the maximum theoretical reversible specific capacity that can be achieved with lithium-intercalated natural crystalline flake graphite is 372 mAh/g. This compares with the nearly theoretical performance of the Lac Knife carbon coated spherical graphites of 360 to 365 mAh/g shown in Section 2 of this paper. At this point in time, lithium ion batteries based on graphite anodes have been engineered to operate at near their limit in terms of capacity output per given cell size. Hence, adding silicon to graphite to go beyond the theoretical capacity limit in lithium ion batteries is gaining significant attention.

With pure silicon, theoretical reversible capacities of 4,200 mAh/g are possible, which is well over 10 times the capacity of graphite. Although the cost of pure silicon also is an issue, problems associated with the swelling and contraction of pure silicon of as much as 400% during lithiation and de-lithiation reactions in the cell excludes its stand-alone application in battery anodes. In the NATO Science Series book⁶ published in 2006, Vyacheslav Barsukov formulated a principle that the only way for silicon to work would be to have it comprised of very small (nano-sized) particles with very small loading levels preferably dispersed on a much larger-sized carbon carrier matrix. In this way volumetric changes in individual particles of silicon would not cause any appreciable harm to the negative electrode on a macroscopic level. The concept of enhancing the performance of graphite by varying the percent addition of silicon to the graphite while controlling the swelling effect of silicon will provide the battery manufacturer with great flexibility in adjusting the capacity of a cell to meet customer demands.

6.2 Preparation of Silicon Enhanced Lac Knife Spherical Graphite

Lac Knife purified flake was air milled and screened to a maximum particle size of 270 mesh and mixed with nano sized silicon in a glove box under a blanket of argon gas. The amorphous silicon used was produced by vapor deposition of silane and has a particle size of 100 nanometers. A mixture of graphite and 4.5% nano-Silicon was prepared and enclosed in an airtight jar under inert gas. The jar containing the graphite and silicon was placed on rollers for 15 minutes, so the graphite and silicon could properly combine and be used as a pre-mixed composite for further processing.

The premixed composite was then spherodized using a proprietary process which trapped most of the silicon inside the spherical graphite particles. A portion of the premixed composite was saved for further processing and electrochemical testing while the remainder was used to produce the carbon coated spherical graphite containing 4.5 wt % Silicon. The process used to apply the carbon coating is described in Section 2.2. Both the uncoated and carbon coated grades of the spherical graphite were further screened to produce a -450 mesh product used for electrochemical testing.

It is the carbon coating that both results in a reduction of the surface area of the spherical graphite from 11.6 to 2.7 m²/g and protects the silicon from forming silicon carbide at the surface.

6.3 Galvanostatic Curves for Lac Knife Silicon Enhanced Spherical Graphite

The galvanostatic curves presented in Figure 11 show the initial electrochemical data from the first three cycles of a cell containing uncoated spherical Lac Knife graphite with 4.5 wt% silicon added at a C/20 cycling rate.

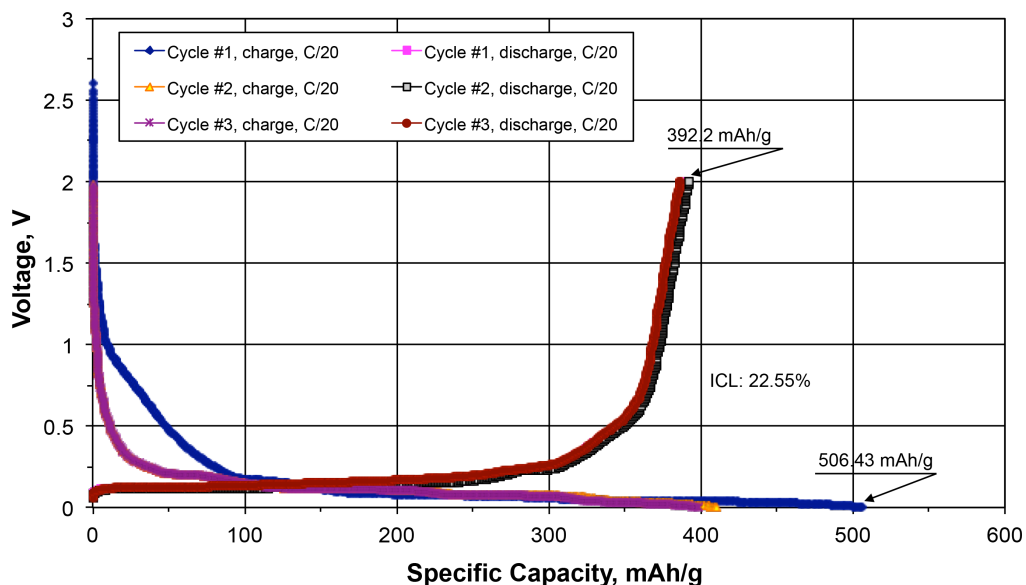


Figure 11 Galvanostatic Charge-Discharge Curves for Uncoated Lac Knife Graphite with 4.5 wt% Silicon Added

The irreversible capacity of the first cycle's charge was 506.43 mAh/g, and the reversible capacity was 392.2 mAh/g. This translates to an irreversible capacity loss (ICL) of 22.55%. Since the three discharge curves are spaced close together and overlap, it is evident that the cycling was exceptionally stable.

Figure 12 presents the galvanostatic curves from three out of the first five cycles of a cell containing carbon coated graphite with 4.5 wt% silicon added at a C/20 cycling rate.

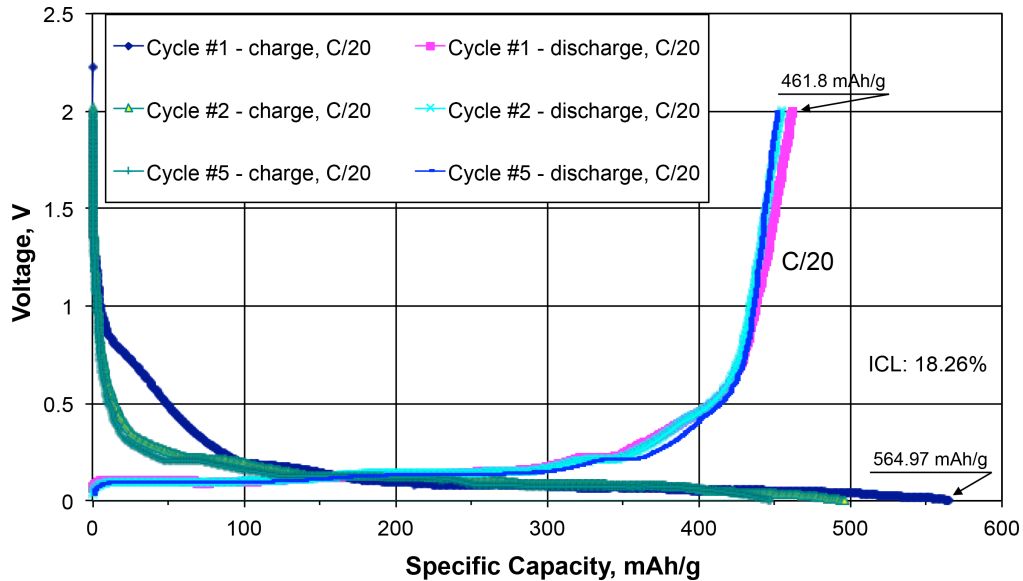


Figure 12 Galvanostatic Charge-Discharge Curves for Coated Lac Knife Graphite with 4.5 wt% Silicon Added

Again, since the discharge curves for the carbon coated spherical graphite are spaced close together, these data also show a highly stable cycling performance. The reversible capacity is 461.8 mAh/g and the irreversible capacity is 564.97 mAh/g, giving an ICL of 18.26%. The ICL is about 5% lower than the ICL of the uncoated material with the same percent silicon added, while the reversible capacity increased. The coated material performed better than the uncoated material because the silicon was better protected by an outside amorphous carbon shell. It also should be noted that the reversible capacity of the carbon coated material is 24% higher than the theoretical capacity that can be achieved with graphite alone.

Enough raw material was left to prepare uncoated spherical graphite with 18% silicon added by the same process that was used in Section 6.2. The galvanostatic curves for this grade are presented in Figure 13.

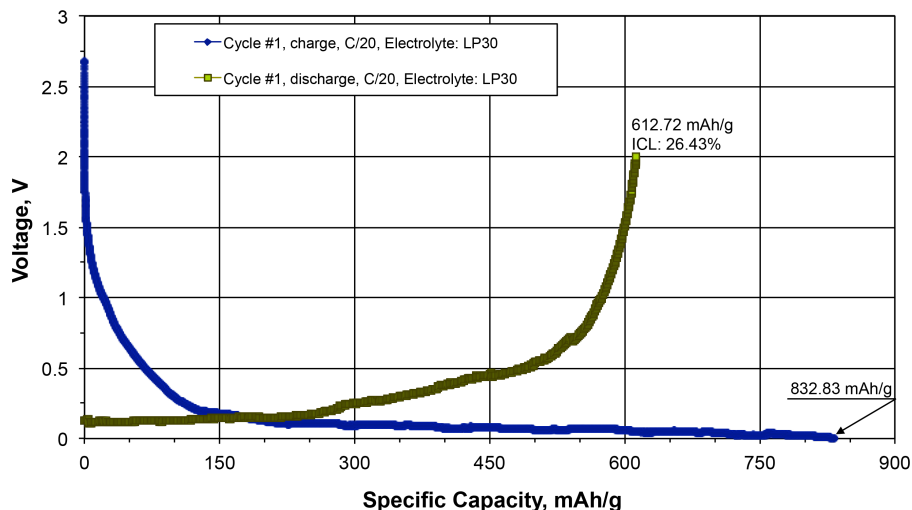


Figure 13: Galvanostatic Charge-Discharge Curves for Uncoated Lac Knife Graphite with 18 wt% Silicon Added

The irreversible capacity of the uncoated spherical graphite with 18% silicon added was 832.83 mAh/g, and

reversible capacity was 612.72 mAh/g, producing an ICL of 26.43%. The reversible capacity achieved is 65% higher than the theoretical capacity that can be achieved with graphite alone and turning the Lac Knife silicon enhanced graphite potentially into a hi-tech, new generation lithium ion battery anode material.

Further work is planned to improve the performance of the silicon enhanced Lac Knife graphite. This will include optimizing the de-aggregation, dispersion and loading of nano-silicon in the graphite matrix, conducting long term cycling tests, improving carbon coating technology for silicon enhanced Lac Knife graphite, determining the best combinations of binder and electrolyte to use and evaluating low cost sources of nano-silicon in lithium ion battery formulations.

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Dr. Doninger has Doctorate, Masters and Bachelor Degrees in Chemical Engineering and is an Honorary Chemistry Professor at Kiev National University of Technologies and Design and authored over 30 technical publications. He previously held positions at International Minerals and Chemicals Corp. and Superior Graphite Co. and joined Focus Graphite as Director of Manufacturing and Technology in 2012 to assist in developing the Lac Knife graphite deposit.



Gary Economo –Biosketch

Gary Economo is President and CEO of Focus Graphite, owner of the high-grade Lac Knife, Quebec flake graphite deposit and co-developer of the Kwyjibo, Quebec rare earths property. Mr. Economo is also co-founder and CEO of Grafoid Inc., a leading graphene R&D, applications development and technology licensing company. Grafoid produces graphene and processes for transformative, industrial-

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