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Towards large-scale silicon nanowire paper fabrication for batteries

Juan J. Vilatela[1,2] , Afshin Pendashteh,[1] Isabel Gómez-Palos[1], Richard Schäufele[1]
[1] Floatech. S.L., Spain
[2] IMDEA Materials Institute, Spain
Juanjose.vilatela@floatech.eu

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ABSTRACT

Si anodes are a central material for the electrification of transport in coming decades, and strategic for Europe given its lack of battery critical raw materials. They can simultaneously increase battery performance and reduce graphite consumption. Si anodes can enable electric vehicle (EV) range near 1000km and charge times below 10 minutes, while reducing pressure of natural graphite, a critical raw material, on a scale of 100s of kilo tonnes per annum (ktpa). In the processes, this could prevent accumulated CO_2 emissions on mega tonne scale during battery manufacture. This presentation introduces a new method for continuous synthesis of a paper-like, freestanding material made up entirely of Silicon nanowires directly from thermocatalytic conversion of silane in a free space reactor, and its application in battery energy storage. It shows progress in the use of the Si nanowire (SiNW) paper as 100% Si anode in Li-ion batteries with high energy density and cyclability, meeting some market entry requirements.

BACKGROUND

Credited for nearly 30% of the reduction in greenhouse gas emissions, batteries are a key technology enabler to mitigate climate change. The lifetime emissions of an electric vehicle are about 80% lower than for an internal combustion engine. Their widespread use in portable electronics, defense and recently in stationary battery energy storage systems is driving battery growth at 20-30 % CAGR. In Europe alone, the accelerated transition towards electric-powered mobility and products requires a 20-fold increase in battery energy storage capacity demand from 2020, reaching around 1000 GWh/a by 2030 in Europe alone (Figure 1).

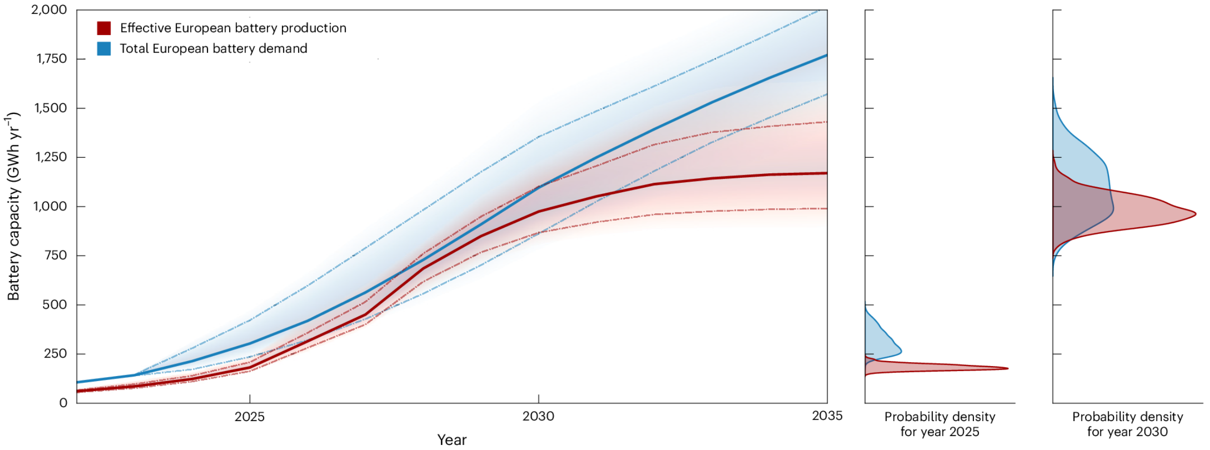

Figure 1. Projected European LIB demand. (1)

This European battery demand will require around 22 million tons per annum of different battery materials (Mtpa) by 2035. However, unlike the solar market which is dominated by a single material (Si), the battery market serves broad power/energy/cost scenarios that thus require different battery chemistries at large scale, each comprising several materials within, with a complex materials interrelation. To simplify, batteries can be visualised as layered devices allowing for multiple different combinations of constituents, but with some dominant ones. For transport and portable electronics lithium-ion batteries (LIB) are set to dominate for the next decades due to their performance. Their energy density is double that of the theoretical limit for sodium-ion batteries, for example.

In LIBs, the most significant upcoming changes are the incorporation of Si in the anode, and the elimination of solvents (in manufacture through dry-processing and in electrolytes in solid-state batteries). Si is a replacement for today's incumbent material, graphite, because of its superior performance, ease of manufacture and abundance. Whereas graphite stores lithium though intercalation of ions between its crystallographic planes ($Li + 6C \rightleftharpoons LiC_6$), silicon undergoes an alloying reaction that can store a much larger amount of Li ($15Li + 4Si \rightleftharpoons Li_{15}Si_4$). These mechanisms, result in Si having an accessible gravimetric capacity almost 10X greater than graphite (3500 mAh/g vs 372 mAh/g).

In addition to its 10-fold higher capacity, silicon is more abundant that graphite. Over 96% of both natural and synthetic battery graphite is produced and controlled by China, making it one of the most pressing critical raw materials (CRM) to replace in Europe and North America. The EU currently extracts less than 0.1% of its total needs. The recurrent introduction of export controls by graphite producers has exposed the sector's vulnerability (2), creating supply risks and potential price shocks.

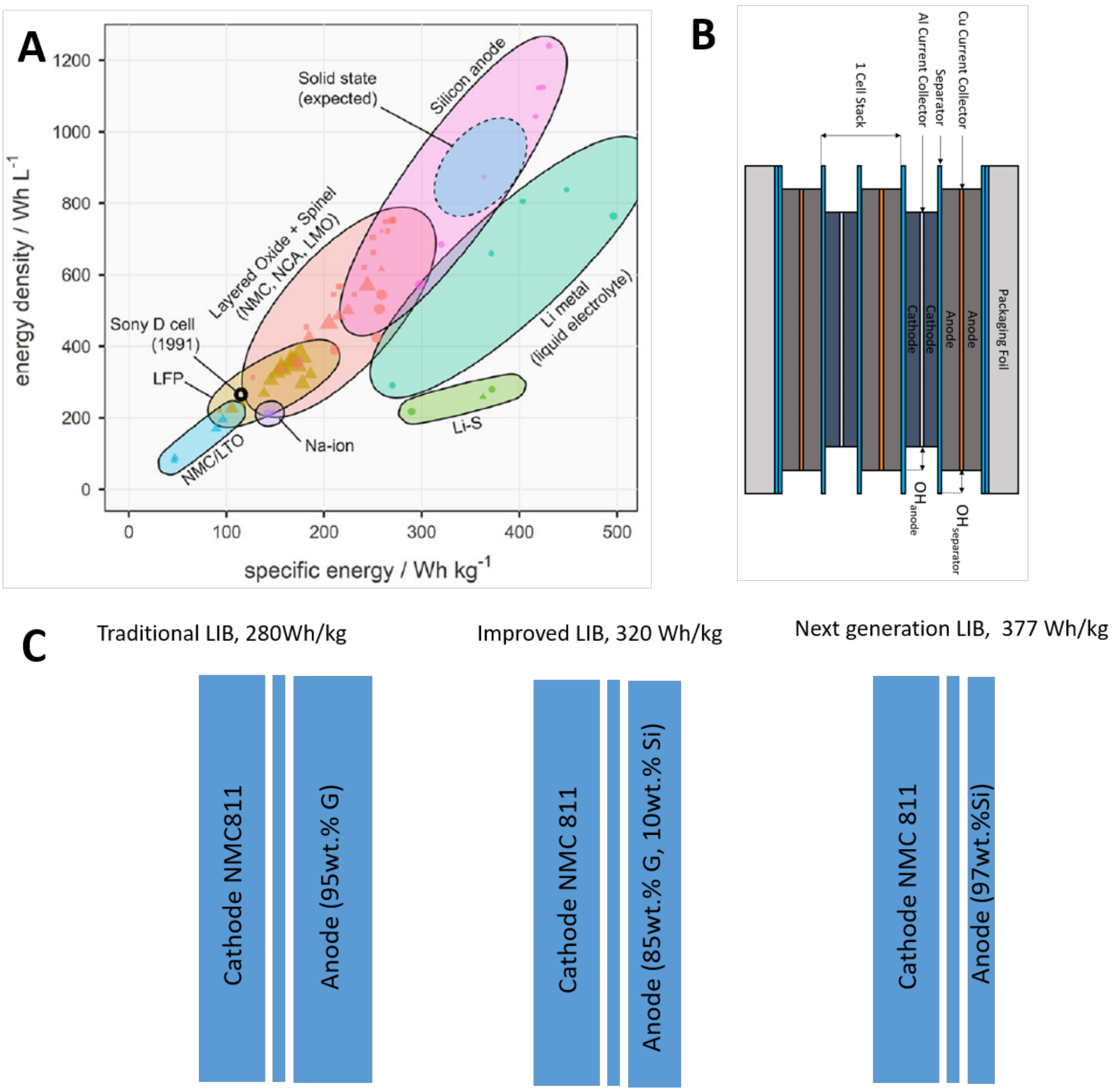

Figure 2. Si anodes for higher performance LIBs. A) Calculated cell energy densities for different battery chemistries. (3) B) Scheme of a prototypical cell (4) and C) comparison of cells with different anodes used in this analysis: conventional G, advanced Si+G, next generation Si. Cell scheme from reference.

TECHNOECONOMICS OF LIBS WITH DIFFERENT ANODES

Improvements in battery cell performance upon introduction of Si depend on multiple cell design parameter, particularly the choice of cathode material and extent of Si utilisation. Figure 2A shows the accessible performance of Si-containing batteries compared to other chemistries. Coupled with a high voltage cathode like Ni-rich layered oxides, at areal capacities above 5mAh/cm^2, full utilisation of an anode of 100% Si leads to cell energy densities above 350 Wh/kg and 1000 Wh/L, amongst the highest for any LIB. They translate into 1000 km and under 10min charge. These values have been recently realised in advanced battery cells starting commercialisation for premium applications (i.e. high performance and high margin). An alternative is to make a blend of graphite and ca. 10wt.% Si as anodes with capacity around 600 mAh/g (5). Blends are used commercially by several cell producers for portable electronics (6) and premium EVs (7).

We carry out a basic top-down technoeconomic analysis of battery silicon to meet 2030 European transport LIB demand. For this, we consider three types of hypothetical 58Ah EV cells: with 100% G, 10% Si & 90% G, and 100% Si, respectively. They are schematically shown in Figure 2B. The cell design is based on the Fraunhofer CellEst model, which enables extraction of realistic cell performance parameters and detailed composition. Their gravimetic energy density is 280 Wh/kg, 320 Wh/kg and 377 Wh/kg, respectively. They represent scenarios of business as usual, incremental improvement and accelerated Si adoption.

Table 1: Comparison of LIB anode technologies

FEATURES	GRAPHITE ANODE	ADVANCED GRAPHITE ANODE (10wt.% Silicon)	REPLACEMENT ANODE 100wt.% Silicon
Anode capacity	372 mAh/g	600 mAh/g	3,100 mAh/g
Cell Energy density	≈240 Wh/kg	>280 Wh/kg	> 350 Wh/kg
Mass anode required by 2030 to meet EU LIBs	550 ktpa	410 ktpa	67 ktpa
References for domestic EU production	1 ktpa natural graphite (2024) Projected 124 kpta synthetic graphite (2030)	1 ktpa natural graphite (2024) Projected 124 kpta synthetic graphite (2030)	400 ktpa Si production (2026)
Estimated price of finished anodes	€12 /kWh	€11/kWh	Under €10/kWh
Estimated embodied emissions levelized by energy stored	9.8 CO_2-eq/$kWh_{storage}$	7.5 CO_2-eq/$kWh_{storage}$	1.2 CO_2-eq/$kWh_{storage}$
Estimated embodied energy levelized by energy stored	66.5 $kWh_{production}$ /$kWh_{storage}$	48.6 $kWh_{production}$ /$kWh_{storage}$	6.6 $kWh_{production}$ /$kWh_{storage}$

For the three battery cell type we estimate the amount of anode required and their projected cost (at 2025 prices), embedded greenhouse gas emissions and embedded energy, levelized by electrochemical performance. The results are summarised in Table 1.

The technoeconomic analysis is carried through combination of four blocks: Si manufacture, cell performance and chemistry, cell manufacturing costs at EV scale, and projected European anode LIB demand, under these assumptions:

Si manufacture. Embedded emissions taken as 10 kg $CO_{2\text{-eq}}$/kg for graphite (average of natural 6 kg $CO_{2\text{-eq}}$/kg and synthetic 14 kg $CO_{2\text{-eq}}$/kg produced in China) (8), and 13 kg $CO_{2\text{-eq}}$/kg for metallurgical Si from China (6 kg $CO_{2\text{-eq}}$/kg for production in the EEA) (9). Emissions from further processing, either conversion of MG-Si to SiH4, or spheroidization and other treatments to make battery graphite, are assumed to be small compared to the initial chemical step and not included.

Cell performance and chemistry. The cell gravimetric energy density and full materials composition is calculated using the CellEst model for a 58Ah EV-type cell (10). The choice and areal capacity of the cathode are key parameters, as the cathode is the limiting electrode. We consider an NMC811 cathode with a high areal capacity of 6mAh/cm^2, in line with the trend of continuous increase in areal capacity communicated to Floatech by cell producers and OEMs. The choice of areal capacity explains the discrepancy with similar analysis done by others (11).
Cell manufacturing costs at EV scale. Detailed manufacturing costs (OPEX, CAPEX) are from the BatPac database of Argonne National Laboratory, which models costing for a 50GWh EV battery plant. The main information extracted is the cost distribution in manufacture of finished anodes of graphite, include the various stages of slurry preparation, coating, drying, etc.
Projected European anode demand for LIBs. European anode demand and the required mass of Si and/or G are calculated considering the projected increase in LIB demand, assuming 75% of LIB demand is for transport and Europe accounting for 33% of global Si market. Energy demand is converted into anode and materials demand data from the cell performance and chemistry module. European LIB demand is from references (12).

As seen in the table, European demand can be met with 67kpta of 100% Si, instead of 410 kpta of blends of 550kpta of G. For reference, we also include values for the domestic production of battery graphite and metallurgical Si. This shows that Europe produces 1/500 of its graphite demand, but nearly 10-fold more Si than needed for LIBs. Therefore, despite increased performance, blends of Si with graphite carry supply risks. In a blend, 1kg of Si requires 9kg of graphite. As a full anode, 1kg of Si replaces 10kg of graphite.

Because of its much higher capacity, the estimated levelized cost is approximately €10/kWh, which is below that of graphite. Similarly, the embedded emissions and energy are both nearly an order of magnitude lower for Si. The embedded emissions of anodes of 100% Si levelized by energy stored, are about 1/8 of those of anodes of graphite (Table 1).

Si ANODE PRODUCTION

Estimates of Si used in anodes are shown in Figure 3. It shows that Si went from marginal to kpta production in a few years. Most of today's capacity is for blends, produced from pyrolysis of monosilane. However, there is rapid progress in the development of new methods to manufacture Si-dominant anodes (>50wt.%) by a handful of startups and metallurgical Si producers. These companies are differently positioned along the production chain: from anode active material to anode production, to cell fabrication. The different manufacturing methods proposed include thin film deposition, crystal etching, microparticle jet-milling and thermocatalytic growth. The prevalent challenge for all these technologies is to demonstrate scalable, cost-effective manufacture of products that can be integrated in the next generation of batteries for widespread use. This landscape shows a clear trade-off: production at relatively large scale (tpa) is demonstrated for active material particles as an irregular powder, which thus need extensive postprocessing to make inks, slurries and anodes prior to cell integration. Alternative new methods apply direct fabrication of finished anodes from monosilane, enabling seamless integration of anodes in cells without any need for solvents, binders, etc. Yet, these disruptive alternative methods have currently lower production capacity.

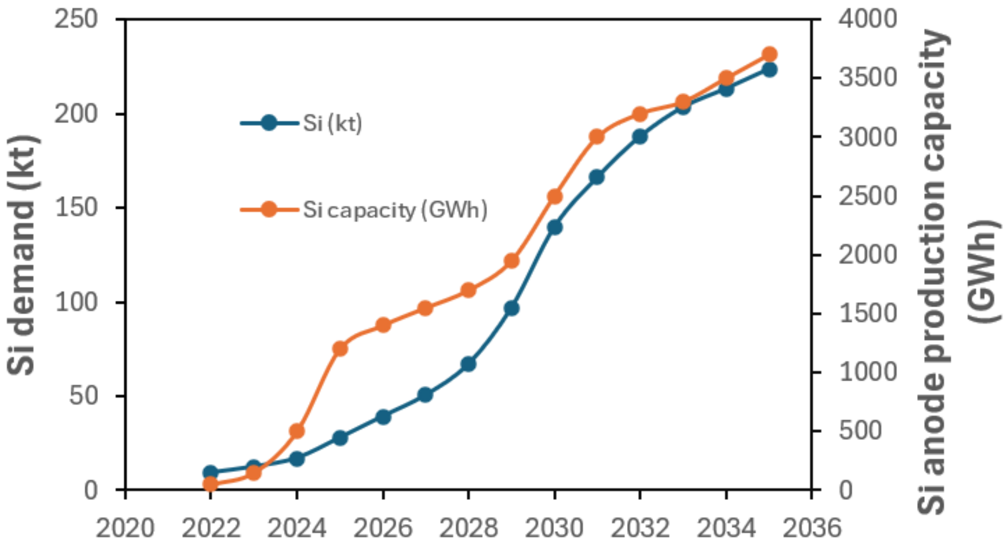

Figure 3. Estimated production of Si for LIBs, based on calculations adapted from Benchmark mineral intelligence report on Si anode production capacity and Si-based electrode market share forecast and other sources. A 10% Si content in composite electrodes is assumed, and an equivalence of 6.4 GWh/kt for Si considering highly optimised cells.

THE FLOATECH PROCESS: SILICON PAPER FROM FREE SPACE REACTORS

Floatech's manufacturing process consists in the thermocatalytic conversion of silane ($SiH_{4(gas)}$) as a precursor into a Si paper consisting of porous network of entangled nanowires. It is a single-step process, carried out continuously in a flow-through reactor. As a separate downstream processing step, the paper-like Si material collected at the reactor outlet is mechanically attached onto a copper foil (current collector used in batteries) to make a finished anode, which is then supplied to a battery producer to assemble battery cells. The manufacturing process is schematically shown in Figure 4.

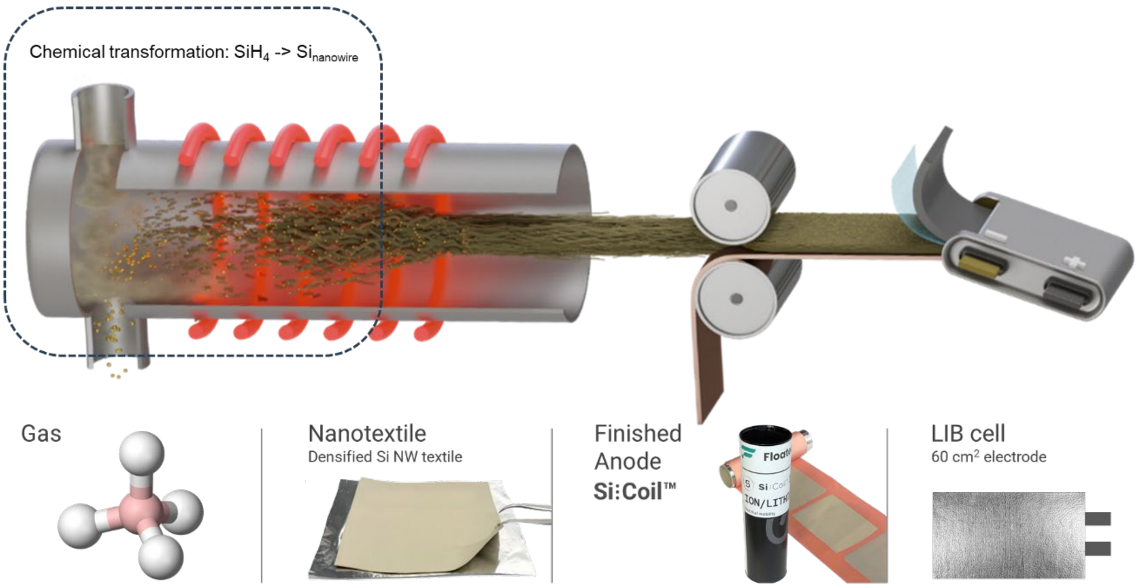

Figure 4. Overview of Floatech's manufacturing process whereby silane is thermocatalytically converted into a freestanding Si paper made of Si nanowires, which is then mechanically attached to a current collector to make a finished anode for battery cell assembly.

The key stage in the manufacturing process is the chemical transformation of silane into Si nanowires (SiNW), monocrystalline Si wires with nano-sized diameter and an aspect ratio of 55. Currently, the process is carried out by flowing reactants and gases through a hot wall tubular reactor at 650 °C. In the process a mixture of silane, a carrier gas and a catalyst aerosol have a residence time under 10s in the hot zone.

When the reactants approach the hottest part of the profile SiH_4 reacts to form an alloy with the metal nanoparticle (e.g. Si-Au). As the alloy particles supersaturate, a nanowire crystal layer nucleates, and growth of the nanowire continues provided Si incorporation into the alloy is maintained. This is known as the vapour-solid-liquid VLS mechanism. The metal nanoparticle acts as reservoir for Si, which is different from a traditional catalysts in that the nanoparticle undergoes a large chemical change during the reaction. It remains incorporated in the SiNWs.

Growth of the nanowire from the alloy particle occurs at very high rates, of the order of ~10-100 µm/s or 10^7 atoms/s, in a regime where the rate-limiting process is Si incorporation into the alloy and growth termination occurs through silane depletion. The nanowires have a diameter distribution reminiscent of that of the metal nanoparticles, and a log-normal length distribution with a mean of 1.5 µm. Because of their high aspect ratio and high concentration, the nanowires form an aerosol that evolves to form large agglomerates through diffusion-limited cluster aggregation (13) within the reaction tube and later super-agglomerates similar to flakes, visible by naked eye.

As a chemical route, Floatech's FCCVD method has a close resemblance to the free-space reactor (FSR) process for Si nanoparticle (SiNP) production. In FSR, Si nanoparticles are produced through nucleation from monosilane pyrolysis in the gas phase. The method was amply studied over the last decades. Through heat management and controlled precursor injection to prevent wall deposition, FSR monosilane conversion into SiNPs can reach over 99% and be scaled up to the industrial pilot level at hundreds of tpa (14). Work is in progress to demonstrate a similar scale-up path for SiNW growth in FSR.

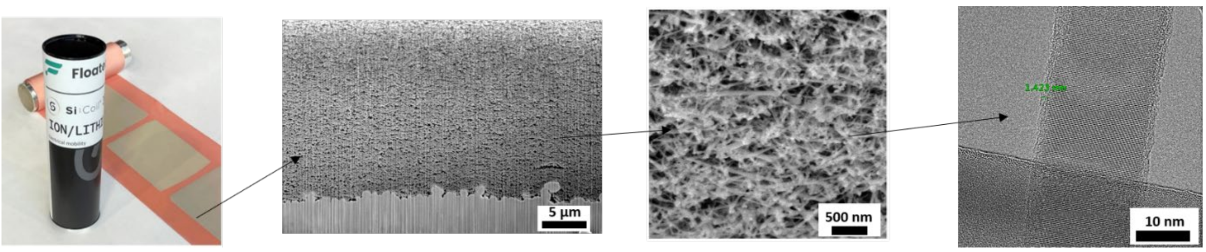

Figure 5. Paper-like silicon comprised of an interconnected network of Si nanowires. Photograph and cross-sectional electron micrographs of a finished anode before cell cycling.

Downstream of the reactor outlet SiNWs are collected as an aerogel on a conveyor belt. Upon mechanical densification the aerogel converts to a SiNW paper. The SiNW macroscopic material is freestanding, flexible and has tensile mechanical properties analogous to those of office paper (15). An example of a finished anode of SiNW on a Cu current collector is shown in Figure 5. The electron micrographs (before cell cycling) show the interconnected network of nanowires and engineered porosity to supress cell expansion.

The SiNW anode can be directly integrated in a LIB cell. Its second cycle reversible gravimetric capacity is over 3000 mAh/g, depending on cell design and test conditions. Anode thickness can be easily adjusted during manufacture in the range 0.1 – 10 mAh/cm^2 (16). By way of example of performance in full cell, Figure 6 shows the capacity retention in a pouch cell with NMC cathode, SiNW anode and solid electrolyte. The N/P ratio is such that the cell is tested under nearly full Si utilization. This implies that the capacity retention is amongst the highest reported, meeting many market specifications and demonstrating that the full use of Si anodes to replace graphite in the next generation of LIBs is feasible and advancing at rapid pace.

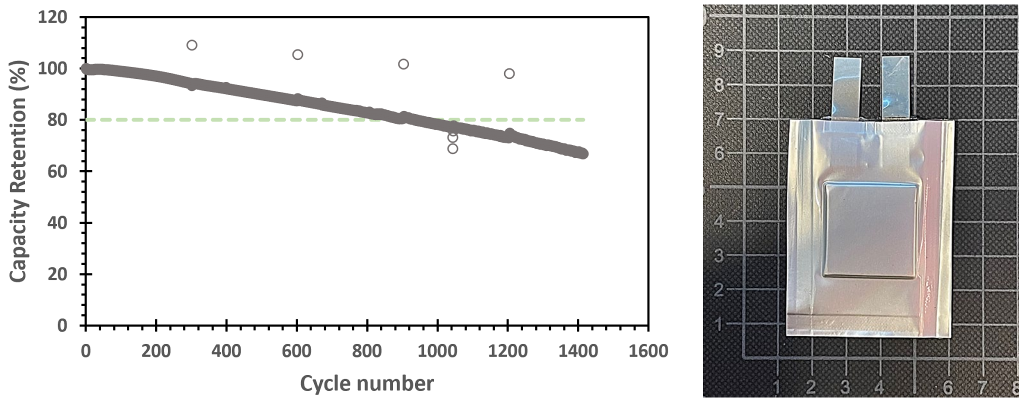

Figure 6. Electrochemical properties of battery cells with Floatech's Si anodes. Capacity retention of full cell with solid electrolyte. (17)

CONCLUSIONS

Silicon anodes are a key material to enable deployment of LIBs for electrification of transport and potentially for stationary storage in battery energy storage systems (BESS). For Europe, Si anodes offer an opportunity to meet battery demand with domestic production, while reducing embedded GHG emissions in anode manufacture.
Around 10% of Si is already blended with graphite to make improved anodes, used in commercial cells for portable electronics and premium EVs. Complete replacement of graphite with anodes of 100% Si will decrease battery time-to-charge and weight, while reducing external supply risks.
There are increasing demonstrations of prototype battery cells with 100% Si that meet market specifications. An example is shown of a solid-state battery with Floatech's SiNW anode reaching over 1000 deep cycles with more than 80% capacity retention, meeting one of the longstanding targets for EVs.
After performance demonstration, for producers of Si anodes the next challenge is scale-up to meet the exponentially growing demand, currently at ktpa and expected to reach Mtpa in ten years.

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