Patents Act 1977 **Opinion Number** **03/18**

OPINION UNDER SECTION 74A

Patent	EP 2194596
Proprietor(s)	NEXEON LIMITED
Exclusive Licensee	
Requester	OneD Material LLC
Observer(s)	NEXEON LIMITED
Date Opinion issued	29 May 2018

The request

1. The comptroller has been requested to issue an opinion as to whether EP 2194596 B1 ("the Patent") is valid under section 74A of the Patents Act 1977 in terms of the following:

   i. the specification of the Patent discloses the invention clearly enough and completely enough for it to be performed by a person skilled in the art;
   ii. the matter disclosed in the specification of the Patent extends beyond that disclosed in the application for the Patent as filed; and
   iii. the subject matter of all of the claims of the Patent is new and hence these claims define a patentable invention.

2. To accompany this request the Requester has provided the following documents:

Ref.	Literature Details
X1	WO 2009/010758 A2: publication of the parent application as filed under PCT/GB2008/002452
X2	Declaration by Professor Kurt W. Kolasinski, dated December 29, 2016
X3	WO 03/011251 A1, published on February 13, 2003
X4	Gao, B.; Sinha, S.; Zhou, O. *"Alloy formation in nanostructured silicon"*, J. Adv. Mater., 13 (11), 5 June 2001, 816-819

X5 Sinha, S., Gao, B. & Zhou, O. "Synthesis of Silicon nanowires and novel nano-dendrite structures". J. Nanoparticle Research, 6 (2004) 421-425

X6 *Kim, J.W.; Ryu, J.H.; Lee, K.T.; Oh, S.M. "Improvement of silicon powder negative electrodes by copper electroless deposition for lithium secondary batteries". J. Power Sources, 147, (1-2) 9 September 2005, 227-233*

X7 US 2007/0087268 A1 published on April 19, 2007

X8 KR 10-2007-0041900 published on April 20, 2007

X9 US 2004/166319 A1, published on August 26, 2004

X10 WO 2007/037787 A1, published on April 5, 2007

X11 US 2004/0214085 A1, published on October 28, 2004

X12 WO 2013/128201 A1, international application by the proprietor
published on September 6, 2013

X13 WO 2015/008093 A1, international application by the proprietor
published on 22 January, 2015

Observations

3. Observations on behalf of the proprietor were received from Kilburn & Strode LLP on behalf of the Patentee on 9 February 2018. These included the following documents in evidence:

   Declaration by Professor. Leigh Canham, dated October 21, 2016.

   Declaration by Professor. Leigh Canham, dated November 14, 2017.

   Experimental report by Dr. Jianmin Wu.

   Translation of the experimental report by Dr. Jianmin Wu, dated October 17, 2017.

   Declaration by Professor Mino Green, dated April 20, 2011.

Observations in reply

4. In response observations in reply were filed by Marks & Clerk LLP on behalf of the Requester on 22 February 2018. Further pieces of evidence were provided:

X14 Liu, Y.; Xiong, Z.H.; Liu, Y.; Xu, S.H.; Liu, X.B.; Ding, X.M.; Hou, X.Y. *"A novel method of fabricating porous silicon material: ultrasonically enhanced anodic electrochemical etching"*, Solid State Communications, 127(2003) 583-588.

X15 Green, M.; Garcia-Parajo, F.; Khaleque, F.; Murray, R.; *"Quantum pillar structures on n+ gallium arsenide fabricated using 'natural, lithography"*, Appl. Phys. Lett. 1993, 62, 264-266.

X16 Green, M.; Tsuchiya, S. *"Mesoscopic hemisphere arrays for use as resist in solid state structure fabrication"*, J. Vac. Sci. Technol.B, 1999, 17, 2074-2083.

X17 Declaration by Professor Kurt W. Kolasinski, dated February 21, 2018

X18 Sinha, S.; Gao, B.; Zhou, O. "Synthesis of silicon nanowires and novel nano-dendrite structures", AIP Conference Proceedings 544, 431 (2000).

X19 USPTO office action relating to US 12/669,216 (Mino Green).

The Patent

5. The Patent was filed on 17 July 2008 with an earliest date of 17 July 2007. It is one of a number of patents/applications stemming from the application published as WO 2009/010758 (document X1). The Patent was published on 9 June 2010 and was granted on 24 October 2012. It remains in force.

6. The Patent relates to a silicon-comprising particle that can be used as an active anode material in a lithium-ion rechargeable battery cell. It seeks to overcome the problem of capacity loss over the required number of charge/discharge cycles arising from partial mechanical isolation of the silicon powder, by providing a silicon-based particle that has pillars extending all over the surface of its core.

7. The Patent as granted has 17 claims of which product claim 1 and method claim 14 are the independent claims.

8. Claim 1 reads:

> *A particle for an electrode comprising silicon, the particle having a silicon-comprising particle core and a plurality of silicon-comprising pillars extending all over the surface of the particle core, wherein the pillars are electrochemically active.*

9. Claim 14 reads:

> *A method of forming a particle for an electrode, the method comprising etching a silicon-comprising particle to form a silicon-comprising particle core and a plurality of silicon-comprising pillars extending therefrom, wherein the pillars are electrochemically active.*

10. All the claims are listed in Annexe 1.

Admissibility of the Request

11. Rule 94(1)(b) of the Patents Rules 2007 provides that:

> *The comptroller shall not issue an opinion if the question upon which the opinion is sought appears to him to have been sufficiently considered in any relevant proceedings.*

12. Whilst much of this evidence was raised as part of opposition proceedings started against EP 2533331, the application that became EP 2533331 was divided from the application (EP 10002163.3) that was granted as the Patent now being considered. These are clearly separate applications, where the evidence provided was put forward in relation to the divisional (i.e. after and not before division). Therefore I believe the Requester is right to argue that the evidence in this request "was not considered during the examination of EP 10002163 and hence the submissions made … are not a mere repetition of arguments that have already been considered pre-grant". I also note that the Observer does not object to the request (instead merely noting that the request for revocation of EP 2533331 was not due to the objections made in the opposition). Whilst I am also happy to consider documents X14-X17 of the further evidence provided in the observations in reply as I do not believe this evidence introduces a new line of argument, I will only consider X18 as far as it demonstrates the commonality of X4 and X5 and not as a further potentially novelty-destroying document, and on balance I believe I cannot consider document X19 as the Observer has no further opportunity to comment on the relevance of this document. The Requester had an opportunity to provide this earlier if they felt that evidence might be required to support the view that X7/X8 were enabling, but did not choose to do so.

The Law

13. Section 1(1) of the Act reads:

> *A patent may be granted only for an invention in respect of which the following conditions are satisfied, that is to say –*
> *(a) the invention is new; …*

14. Section 14(3) of the Act states:

> *The specification of an application shall disclose the invention in a manner which is clear enough and complete enough for the invention to be performed by a person skilled in the art.*

15. Section 76(2) of the Act states:

> *No amendment of an application for a patent shall be allowed under section 15A(6), 18(3) or 19(1) if it results in the application disclosing matter extending beyond that disclosed in the application as filed.*

16. The test for added matter set out by Aldous J in *Bonzel and Schneider v Intervention Ltd*[1] is as follows:

> 1) to ascertain through the eyes of the skilled addressee what is disclosed both explicitly and implicitly in the application;
>
> 2) to do the same in respect of the patent as granted;
>
> 3) to compare the two disclosures and decide whether any subject matter relevant to the invention has been added whether by deletion or addition. The comparison is strict in the sense that subject matter will be added unless such matter is clearly and unambiguously disclosed in the application either explicitly or implicitly.
>
> This was summarised by Jacob J in *Richardson-Vicks Inc.'s Patent*[2] as:
>
> *"the test of added matter is whether a skilled man would, upon looking at the amended specification, learn anything about the invention which he could not learn from the unamended specification."*

Claim construction

17. Firstly I need to construe the independent claims 1 and 14 of the Patent, that is to say I must interpret them in the light of the description and drawings as instructed by Section 125(1) of the Act. In doing so I must interpret the claims in context through the eyes of the person skilled in the art. Ultimately the question is what the person skilled in the art would have understood the patentee to be using the language of the claims to mean. This approach has

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[1] *Bonzel and Schneider. v Intervention Ltd* [1991] RPC 553
[2] *Richardson-Vicks Inc.'s Patent* [1995] RPC 568

been confirmed in the recent decisions of the High Court in *Mylan v Yeda*[3] and the Court of Appeal in *Actavis v ICOS*.[4]

18. Both the Requester and the Observer agree that an important construction issue is what is meant by "pillar" in both these claims. The Requester asserts in the initial request that the term pillar is routinely used within the art to define an elongate nanostructure and the skilled addressee would consider it interchangeable with other commonly-used terms for such nanostructures like nanorod or nanowire. In the observations in reply they go further by stating that structures with a small aspect ratio (height divided by diameter) have been called pillars, pointing to documents X15 and X16, which disclose aspect ratios of 0.5, and describe pillars with "flat tops and sharp tops" respectively. Therefore they argue that the skilled addressee would have understood that the term pillars may be used to define a structure that can have an aspect ratio of less than one and either straight or tapered walls, and that this definition was routinely used by at least one of the named inventors of the Patent.

19. The Observer notes that the Requester has argued that "pores and pillars are essentially complementary features in the sense that 'pores' are the voids in the Si material, and the Si material between the voids forms the 'pillars'" (this drawing on the text of X12 page 7 line 21-page 8 line 2). The Observer suggests that terms used in X12 are irrelevant as this document was published over 6 years after the priority date of the Patent. I agree with this statement, it is important that the term is construed as it would have been at the priority date of the Patent.

20. Furthermore I have read the declarations from Professors Kurt W. Kolasinski and Leigh Canham and their positions on what would be considered a pillar in the art at the priority date. There is obviously some disagreement and thus, having weighed up the contributions carefully and being mindful of the absence of a unified view on this matter, I feel I must concentrate on the Patent to see what the skilled worker would think the Patentee intended the scope of 'pillar' to be.

21. The Patent itself states at paragraph 0017:

> *"The pillars may be regular or irregular. The pillars of the present invention may be 0.08 to 0.70 microns in one dimension, preferably 0.1 to 0.5 microns, more preferably 0.2 to 0.4 microns, and most preferably 0.3 microns or above. In a second dimension, the pillars may be 4 to 100 microns, preferably 10 to 80 microns, more preferably 30 microns or above. The pillars thus may have an aspect ratio of greater than 20:1. The pillars may have a substantially circular cross-section or a substantially non-circular cross-section."*

22. This statement causes a minor problem as the pillars do not have to be circular

[3] *Generics UK Ltd (t/a Mylan) v Yeda Research and Development Co. Ltd & Anor* [2017] EWHC 2629 (Pat)
[4] *Actavis Group & Ors v ICOS Corp & Eli Lilly & Co.* [2017] EWCA Civ 1671

in cross-section and thus, whilst it might be reasonable to assume that one dimension quoted is the depth of the pillar, there is no clear statement that the longest dimension is the depth. These dimensions could be read as two dimensions of the cross-section. However on balance I conclude that the skilled worker would understand the longer dimension to be the depth as the first and second dimensions cannot be the same as would be necessary for a pillar of circular cross-section. Nonetheless the dimensions here are only preferred features rather than necessary features of the pillars defined in claims 1 and 14.

23. Thus the skilled worker is left with "pillars" that are "regular or irregular" and that "may have a substantially circular cross-section or a substantially non-circular cross-section." The dimensions given in the Patent are not limiting, but they suggest that the Requester's initial comments regarding pillars being "elongate nanostructures" are reasonable. My view in this regard is reinforced by the statement at paragraph 0027 of the Patent which reads "In particular it is believed that the structure of the particles that make up the composite overcomes the problem of charge/discharge capacity loss. By providing a particle with a plurality of elongate or long thin pillars the problem of charge/discharge capacity loss is reduced". Therefore I do not think that a structure with an aspect ratio (height divided by diameter) of one or less can be sensibly regarded as a pillar (the use of the term "pillar" in document X15 referred to above notwithstanding) given the thrust of this latter passage.

24. Secondly the intended meaning of "all over the surface of the particle core" must be construed. The Requester states "[t]he phrase 'all over the surface of the particle core' does not appear to define that the pillars must cover 100% of the particle core, since claim 2 restricts pillar coverage to 10-50% of the surface of the particle core and the subject matter of Claim 2 must be covered by Claim 1. Accordingly the phrase 'all over the surface of the particle core' must mean that the pillars are in some way distributed over the whole surface area of the particle core, but the pillars do not necessarily have to cover 100% of the surface of the particle core". Before going on to argue "[h]owever, there is a certain conflict between the language used in these two claims and the expression 'all over' is somewhat colloquial, so the precise ambit of this expression is unclear and impossible to interpret with any certainty".

25. In response the Observer questions why having come to a logical conclusion as to what the phrase "must mean", the Requester then considers interpretation of the phrase to be impossible. In their observations in reply the Requester states that:

> "Just because the expression 'all over the surface of the particle core' can be interpreted to mean that the pillars are in some way distributed over the whole surface of the particle core, it does not mean that Claim 1 is sufficiently clear. The key question is the nature of the distribution. The skilled reader must be able to understand when this condition is met and when it is not. … Given that Claim 1 is clearly intended to cover a particle having a sparse distribution of pillars (10%, for example, in view of the dependency of Claim 2), it is impossible for the reader of Claim 1 to understand the nature of the distribution that is meant by 'all over',

because a particle having 90% of its surface unoccupied does not accord with any normal meaning of the expression."

26. I am afraid that in this situation I struggle to see the problem presented by the Requester. The "normal meaning of the expression" or how "colloquial" is the term are not relevant to determining what the skilled worker would think the patentee meant in view of the contents of the description. As has been suggested "all over" cannot mean 100% coverage is required both because of claim 2 and more importantly because the method taught for generating these particles, etching, necessarily results in coverage of less than 100%. Secondly, the method taught for working the invention does not control the position of nucleation i.e. nucleation does not occur on only a given face, as it might do if the same method were used on a silicon wafer for example. Therefore in this context I conclude that "all over" equates to a situation where the distribution of pillars over the surface of the core is unrestricted and across all of the core – it indicates distribution not density.

27. I also note at this point that both parties appear to agree that the "silicon-comprising particle core" and the "plurality of silicon-comprising pillars" as defined in the claims do not necessarily have to be made of the same material. I agree that this is an appropriate construction.

Sufficiency

28. The Requester suggests that the notional person skilled in the art for the purposes of sufficiency is "one skilled in the fabrication and application of battery electrode materials. Such a person would have a knowledge of the various materials conventionally used for battery electrodes and would be aware of the advantages and disadvantages of a silicon-based material relative to other materials, such as graphite-based materials." The Observer has proposed no alternative skilled person and I agree that this is a reasonable definition.

29. I will now consider the three generally accepted aspects of sufficiency separately.

Classical insufficiency

30. The Requester asserts that the application is classically insufficient on the basis that the Patent only teaches a single method for forming the claimed pillared particles using "chemical galvanic exchange" (also known as MACE, metal assisted chemical etching) and that the mechanism by which this is suggested as working in the Patent is "technically and factually incorrect". They further suggest that the disclosure of X13 (WO 2015/008093) which states that "using this method, it is not possible to control the placement of catalyst particles and thus the size, spacing and arrangement of the surface features cannot be controlled", establishes that the method cannot be worked without exercising any invention or any prolonged research, inquiry or experiment. However, the request includes no explicit statement going as far as to suggest that the figures shown in the Patent do not show particles falling within the scope of the claims

or that these figures do not show particles formed by the MACE method.

31. The Observer suggests that "the underlying mechanism by which the etching proceeds is, in fact, irrelevant to the question of sufficiency" as the method described at pages 14-17 is a method for performing the invention as evidenced by what is shown in Figure 2 described as "an electron micrograph of a pillared particle according to embodiments of the present invention" at paragraph 0026 of the Patent. I agree with this. The method set out in the application as filed includes a nucleation step carried out by adding particles to a solution of hydrogen fluoride and silver nitrate in ethanol and water at room temperature with the reaction stated as taking 7-10 minutes for the stated particle size and a shorter time if smaller particles are used. I am not in a position to dispute the veracity of the statement about figure 2, and the method of nucleation described appears straightforward and requires no particular control beyond varying the time period for the reaction. An understanding of the mechanism is not required to work the invention as no active steps are being taken to control how the surface of the particles is covered in silver beyond concentration of the solution, amount of ethanol present and length of reaction. These would all appear to be within the ambit of the skilled worker who for sufficiency purposes is considered to be seeking to work the invention. Accordingly, I conclude that the Patent does provide a method of working the invention and that the invention of both claims 1 and 14 is classically sufficient.

32. The Requester made a further argument stating in the observations in reply that "[t]he Observer has not substantiated their claim that the person of skill in the art could use the teaching of the patent in combination with common general knowledge at the priority date to vary the height, aspect ratio and coverage of the pillars produced using the method set-out in the patent." This is a line of argument that in my view relates to insufficiency by excessive claim breadth rather than classical insufficiency and thus I will not consider it further here.

Insufficiency by ambiguity

33. The Requester argues that the phrase "all over the surface of the particle core" defies clear interpretation in view of claim 2. I have already discussed this in the claim construction section and concluded that on balance it does not provide a problem and thus I am content that the claims, specifically claims 1 and 16, are not insufficient by ambiguity.

34. In addition the Requester suggests that the wording of dependent claim 8 is insufficient in the same way because the definition "compris[ing] n-type or p-type silicon" results in the claim being defined in terms of a feature which could not be determined by the skilled worker utilising metallurgical grade Si. In response the Observer makes various arguments. The most pertinent of these are that the claim is not limited to metallurgical grade silicon and moreover that the use of the word "comprise" is not exhaustive and so a particle formed of silicon with both n-type and p-type silicon will still fall within the scope of the claim. I agree that the claim is somewhat less limiting than on first inspection and do not believe that this wording brings about insufficiency through

ambiguity.

Insufficiency by excessive claim breadth

35. The Requester has put forward the argument that claim 1 of the Patent covers two types of particle: one being a particle in which the pillars are made of a material different from the material of the particle core; and the other being a particle in which the pillars are made from the same material as the particle core.

36. The Requester then asserts that "the sole information given in the Patent for forming the pillars is to etch a silicon-comprising particle. As a result of the disclosed etching, the pillars are necessarily made of the same material as the remaining (non-etched) particle core…. In the Patent, there is no indication whatsoever as to why a particle having pillars and core made of different materials would be desirable or as to how such particle could be obtained".

37. It is further argued that since only metal assisted chemical etching (MACE) is exemplified then because the metal deposition, or nucleation step, of MACE requires a metallic or semiconducting particle that the silicon comprising particles cannot be an insulator or feature an insulating layer.

38. The Requester then sets out a number of imaginable ways to approach the problem and their difficulties.

39. In response the Observer notes the lack of evidence provided to back up the assertions made with regard to the problems of the imaginable ways to produce a particle with different core and pillar compositions, and states "we submit that these examples are not actually relevant to the question of sufficiency, even if they could be substantiated." I agree on both points and thus I shall not contemplate these methods as such any further.

40. However the Observer goes on to state that "Nevertheless, we submit that in view of the teaching of the Patent and common general knowledge, the skilled person could, in fact, use at least one of the methods listed by the requester to prepare a pillared particle in which the core and pillars comprise different materials without undue burden…we submit that the list they have provided is not exhaustive and other methods are known to the skilled person that could be used to prepare pillared particles…".

41. In the observations in reply the Requester questions why the Observer refers to other methods which the skilled person could use, but then does not go on to specify them if they would be obviously apparent.

42. At this stage I must also mention the point made by the Requester in the context of classical sufficiency that "[t]he Observer has not substantiated their claim that the person of skill in the art could use the teaching of the patent in combination with common general knowledge at the priority date to vary the height, aspect ratio and coverage of the pillars produced using the method set-out in the patent."

43. Thus, taking the various arguments and counter-arguments, the issues to be considered can be split into: i) is there enablement for a pillared particle where the core and pillars are made of different silicon-comprising materials; and ii) is the whole breadth of claim 1 enabled in relation to particles where core and pillars are of the same material?

44. At this point, considering issue i), I again return to the teaching of the Patent. The Requester is correct to point out that MACE is the only exemplified method and that this as described relies on the core and pillars being of the same material. In addition the only discussion of methods beyond that exemplified is the discussion of "wet etching/using a chemical galvanic exchange method" at paragraph 0029 of the Patent. No other methods are disclosed or suggested. Furthermore I have not been provided with evidence that allows me to conclude that the skilled worker would instead be able to draw on their common general knowledge for suitable methods to work the invention (see paragraphs 38-42 above).

45. The Patent also seems to suggest that the method is associated with a particular advantage. As paragraph 0025 states (my emphasis) "This good reversibility is considered by the present inventors to be due to the ability of the silicon pillars forming part of the structured silicon particle to absorb the volumetric expansion/contraction associated with lithium insertion/extraction from the host silicon without the pillars being broken or destroyed". It is not clear to me whether this advantage would also be applicable to a particle with different materials for core and pillars, and there is no guidance as to how this advantage may be obtained more broadly.

46. Thus, in the absence of any direction regarding methods which might be suitable to fabricate the required particles with the advantage described above, and more importantly in the absence of discussion or exemplification of any methods utilising core and particle made from different materials in the application as filed, I conclude in relation to issue i) that claim 1 represents an invitation to conduct a research programme rather than a claim enabled by the common general knowledge.

47. Furthermore, in relation to issue ii), as noted by the Requester the exemplified method (MACE) only works with semiconducting or metallic material, and also the method taught only allows limited control of height, aspect ratio and coverage of the pillars produced (paragraph 0037 describes what is prepared with silicon powder from Elken of Norway with a pre-etching size of 400 X 300 x 200 µm – "pillars are produced all over the surface having a pillar height of approximately 25 to 30 µm, a diameter of approximately 200 to 500 nm and a pillar surface density , F, of 10-50%, more typically, 30%", but there is no teaching as to how, for example, a pillar surface density of greater than 50% might be achieved). Thus I conclude that claim 1, as far as it relates to pillared particles made of one silicon-comprising material (issue ii)), also cannot be worked across its full breadth on the basis of the teaching of the Patent.

48. Therefore I consider that claim 1 (and claim 14) is insufficient through excessive claim breadth.

49. The Requester has suggested that the dependent claims are all also insufficient. I agree; none of the dependent claims limits the claims to particles where the core and pillars are made of the same material and thus all the independent claims are insufficient by excessive claim breadth for the same reason(s) as the independent claims.

Added matter

50. The Requester argues that matter has been added by intermediate generalisation due to the incorporation of the wording "the particle having a silicon-comprising particle core" into claim 1. The Requester argues that the addition of this statement to claim 1 has resulted in matter being added because the current wording of claim 1 allows for the situation where the core and pillars are both silicon-comprising, but made from different silicon-comprising materials whereas "[t]he feature of the particle core being made from a silicon-comprising material was … only disclosed in the application for the Patent in the context of a method in which the pillars are made from the same silicon-comprising material. Thus the Requester asserts that the skilled addressee is being presented with information that they could not have derived from the application as originally filed.

51. In response the Observer argues that it is clear from the application as filed that the particle core may comprise silicon, irrespective of the method used to make the pillared particle. The Requester points to claims 1 and 9 of the application as filed (together with various other parts of the description) as examples of passages where the core being silicon-comprising is disclosed. In particular the phrase "particle and/or pillars" in claim 9 is relied upon to make it clear that the core may be silicon.

52. Looking at the application as filed it is clear that the particle core may indeed be silicon-comprising. The Requester's argument is however that the amendment introduces the idea that the core and pillars may be of different silicon materials. I do not agree. Paragraph 0015 of the application as filed states "The first aspect of the invention provides a particle comprising silicon having a particle core and an array of silicon-comprising pillars extending therefrom." This is rather odd wording. The teaching of most of the document is clearly that the core and pillars are formed as one piece and thus the whole particle is the same material. This paragraph however makes both the point that the whole comprises silicon and that the pillars are silicon-comprising. This opens up the possibility that the core is not silicon and thus by extension must include the possibility that the core is a different silicon comprising material. However in view of my conclusions with regard to insufficiency through excessive claim breadth my conclusion here is moot.

Novelty

53. I shall concentrate in the first instance on independent claims 1 and 14 in turn; only considering the dependent claims if I find that the independent claims are

not new. I have not been asked to consider the inventiveness of the claims.

54. The Requester suggests that the "attribute of being electrochemically active is observed when pillars are made of silicon". The Observer has not refuted this statement and thus I will not consider this aspect further.

X3

55. The first document brought to my attention by the Requester is X3. At page 22 lines 12-24 Example 4 of X3 describes *"Silicon On Insulator (SOI) wafers with a 30 micron thick Si layer were patterned using a 30 micron square optical mask and HPR-505 photoresist of thickness 1.55 micron, and then dry etched for 24 minutes down to the oxide layer. This generated the array of 30 micron cubes shown in figure 2 (a, b). These particles were then released from the wafer by immersion in HF which dissolves the underlying oxide support. Further size reduction, rounding of corners and porosification is then achieved via stain etching in a solution containing HF, nitric acid and water. Figure 2 (c) shows an example of a 100 micron perfect silicon cube that has been greatly reduced in size and porosified in one etching step using a 50 to 1 volume ratio of 40wt% HF to 70% nitric acid".*

56. The Requester argues that the silicon cube anticipates the claimed particle where the porosified surface shown in figure 2(c) equates to the pillars.

57. The Observer disagrees noting that there is no mention of pillars in X3, and that the figure "shows that the surface features of the porosified cube have a small aspect ratio (approximately 1:1), and, therefore, they are simply not pillars. That is to say, the surface roughness resulting from the porosification of the cube does not equate to a surface pillar extending from the particle core." The Observer further argues that the particle shown in Figure 2c of X3 is so highly etched that even if the surface features are regarded as pillars then the surface features would extend from a porous layer rather than a particle core as required in claim 1.

58. The Requester's observations in reply deal with the last point convincingly in my view in that they note that the claims of the Patent do not require the core to be solid. The Requester further states "[a]s noted by the Observer, the surface features of X3 have an aspect ratio of approximately 1:1. However, this fits with the definition of a "pillar" that was used not only by the inventors but also others within the art at the priority date".

59. In addition both the Requester and the Observer rely on the declarations of Professors Kolasinski and Canham as to whether stain etching can produce pillars within the meaning of the Patent.

60. However, in considering this document I do not think that I need to reach a conclusion as to whether stain etching could or could not produce pillars according to the invention. The level of proof for matters of fact is the balance of probabilities. In X17 Professor Kolasinski states that stain etching can indeed produce pillars, but from the evidence in X3 (figure 2c) I cannot be confident that "pillars" falling within my construction of the term have been

produced or that they would necessarily be produced following the method in X3. Therefore in the absence of a clearer image I cannot say that this document is novelty destroying. Indeed given that both the Requester and Observer appear to agree that the aspect ratio shown in the figure in X3 is 1:1 then, if my construction of 'pillar' requiring an elongate structure is correct, the disclosure would not be an anticipation of claim 1. The same reasoning naturally applies to claim 14.

X4 and X5

61. There was some confusion as to whether both X4 and X5 were being put forward as novelty destroying. The Requester confirms that both are considered to impugn the novelty of "claims 1 and 14", and that both documents relate to the same material. In support of this the Requester also provides X18 stating that X18 has the same content as X5, but includes Figure 1 which is identical to Figure 1a of X4. However, due to this lack of clarity in the original request (only X4 is asserted as being novelty destroying) and the fact that the Observer has not presented arguments specifically in relation to X5, I shall only consider X5 insofar as it supports the explicit and implicit disclosure of X4. Both X4 and X5 relate to n-Si materials fabricated by laser ablation. In particular X4 at page 817 describes *"[t]argets composed of Si powder and 10 at.-% Fe were sintered and ablated by a 532 nm Nd:YAG laser at 1150 ºC under a constant argon flow. Transmission electron microscopy (TEM) examinations showed that the as-synthesised materials contained about 2:1 ratio of nanowires and nanoparticles that are 10-30 nm in diameter (Fig. 1a)*". The Observer questions whether the figure, and by extension the document as a whole, does indeed show nanowires and nanoparticles joined. The Requester points to the statement in X5 in the caption for Figure 3 that *the "SiNWs are protruding out from the nano-cluster"*. This and the statement at page 423 of X5 that *"The long and spaghetti-like silicon nanowires originated from the Si/Fe cluster (nanoparticles)"* make it clear to me that the nanowires and nanoparticles are in fact joined in X5. However, whilst this is apparent from reading X5, it is not demonstrably so on reading X4 in isolation. The Observer's argument appears to me to have merit when only the figure in X4 is available to demonstrate that the clusters and nanowires are linked.

62. X4 discloses silicon-comprising particles and silicon-comprising nanowires. These nanowires are elongate nanostructures with an aspect ratio greater than 1:1 and thus in shape satisfy my construction of "pillars". However in the absence of a clear disclosure that the particles and nanowires are joined on balance I must conclude that claim 1 is novel over this document.

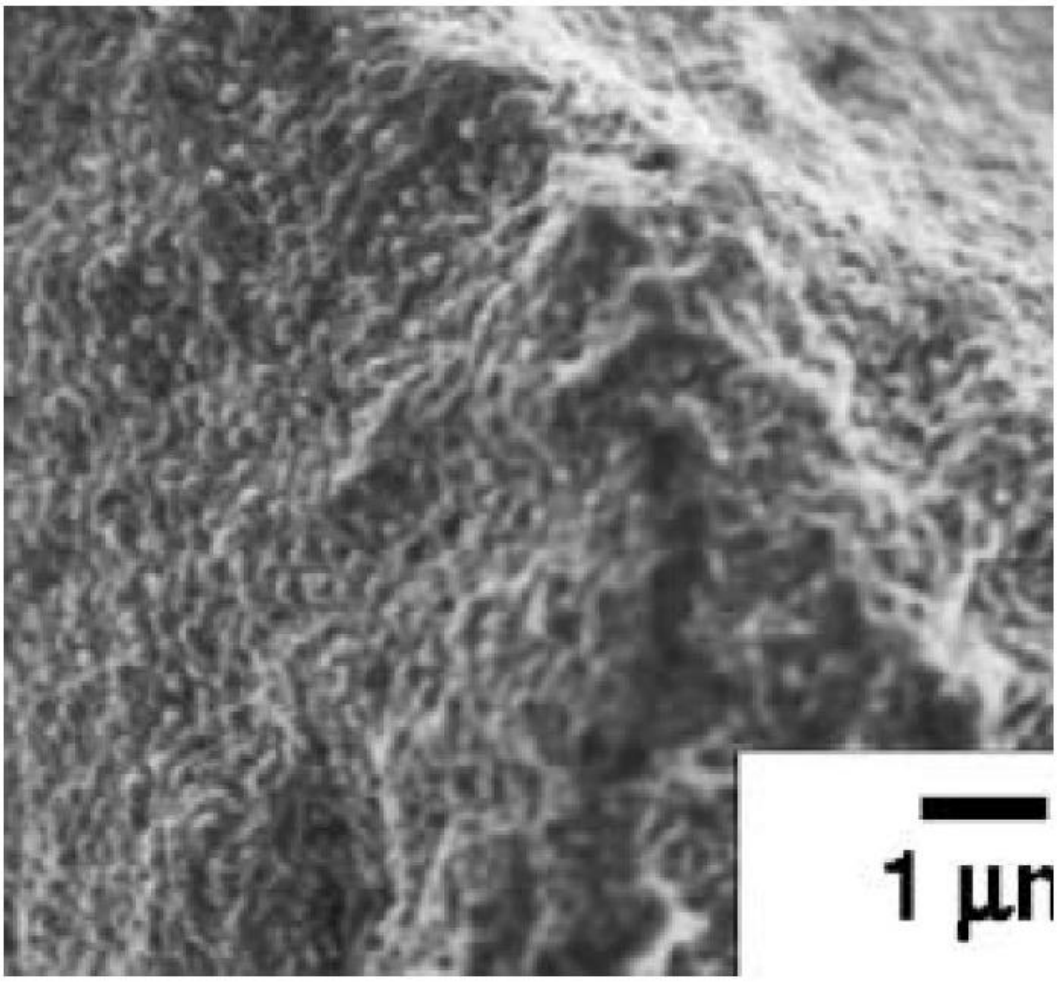
63. Had I considered X5 I would have also had to determine whether these nanowires could be said to be "extending all over the surface of the particle core". I stated in the claim construction section above that I considered this phrase must relate to distribution not density. This being the case, and given that there appears to be no limitation on where on the particles in X5 the nanowires may extend from, I would have concluded that the disclosure of X5 fits this definition.

64. The materials of X4 and X5 are generated by use of laser ablation. As this is

not an etching method I assume that the suggestion in the observations in reply that X4 and X5 show a lack of novelty in claim 14 is in error.

X6

65. Document X6 relates to enhanced electrical conductivity of Si powder for use in anodes in lithium secondary batteries by depositing copper on the silicon surface using "electroless deposition". Section 2.1 of the experimental section of this document makes reference to *"etching Si powder to enhance the surface roughness"*. The Requester asserts that this etching with HNO_3 + HF mixtures to obtain particles (see figure 1(c) of X6) with what is described at page 229 as a *"roughened Si surface with a high population of thorn-like tips"* produces particles falling within the scope of claim 1. The Observer suggests that Figure 1(c) does not show any of these thorn-like tips and that the document does not disclose pillared particles. The Requester subsequently provided a blown-up image of the figure 1(c) (reproduced below) purporting to show the thorn-like tips.



On viewing this image I can certainly see surface roughness, but must confess that I struggle to see that roughness as thorn-like. Moreover I am not convinced on the basis of the evidence provided that what is produced on the surface of

this powder are elongate nanostructures that could be called pillars. Therefore I conclude that claims 1 and 14 are novel over document X6.

X7 and X8

66. The Requester has drawn my attention to paragraphs 0017 and 0018 of document X7 which describe *"an anode active material [which] includes metal core particles, metal nano wires formed on the metal core particles as a single body, pores between the metal core particles and the metal nano wires….In one embodiment, the metal nano wires and the metal core particles in the active anode material may include a metal that can be alloyed with lithium. Nonlimiting examples of suitable materials for the metal nano wires and the metal core particles include Si…*". Example 1 of X7 then sets out a method of the invention utilising Si metal powder. On the face of it this is a novelty destroying disclosure: the method utilises silicon and although the form of material prepared by this method is not stated as a method according to the invention, I take it to afford the claimed "anode active material" where the nano wires are equivalent to the pillars defined in claim 1 of the Patent.

67. However, the Observer suggests that X7 does not provide an enabling disclosure and supplies a declaration from Professor Green to that effect.

68. The observations also state that "during the prosecution of European EP 2 533 332, the European Patent Office were satisfied that X7 is not an enabling disclosure". In reaching a conclusion as to the nature of the disclosure of X7 and X8 I will not take this statement into account. Not only do I not know why the EPO did not pursue this document as a citation in relation to EP 2 533 332 (the file open to public inspection appears to be silent on the reason for not maintaining this objection), but also this application related to a different invention to that being considered in the Patent.

69. Professor Green's declaration includes an attempt to reproduce the method of X7. However the method used differs in two respects from that disclosed in example 1 of X7: i) the molecular weight of the polyvinyl alcohol used is 9000 rather than 500; and ii) omits the second pulverisation step from the method. The declaration states that the last pulverisation step appears unnecessary and this seems reasonable given that the method defined in claim 10 of X7 omits this second pulverisation step. Nonetheless the method described in the declaration differs from that in the example of X7 and thus whilst it can be used to demonstrate that the method of the application does not work across the scope of the invention claimed in that patent application, it does not provide me with evidence that the actual example in X7 is not enabled. On that basis, without further evidence, I can only conclude that example 1 of X7 is enabling and that therefore claim 1 is not novel. If I am wrong and X7 proves to lack an enabling disclosure then of course it would follow that the Patent is novel over X7.

70. The Requester further asserts that claims 12, 13 and 16 are not novel on the basis of X7. Claim 13 defines an electrode with the particles of claim 1 as an active material and thus given that X7 concerns anode active material this claim is also not novel, with the caveats above, on the basis of X7. Claim 12 defines

the dimensions of the particle ('a first dimension of 10 µm to 1 mm'). Example 1 of X7 defines a particle with a starting dimension of less than 43 micrometres before etching. I therefore conclude that the final product will have a similar diameter over the nanowires and thus fall within the scope of claim 12.

71. Before considering dependent claim 16, I must note that although there is no reference to method claim 14 in the request, I assume that this claim is also considered not novel (as claim 16 is dependent on claim 14). Similarly, the Requester also makes reference to figure 2 of X7 in their discussion of claim 2, but does not explicitly state that they consider claim 2 to lack novelty on the basis of X7, and suggests claim 17 lacks novelty without reference to any particular document.

72. Claim 14 defines a method of forming a particle for an electrode comprising etching. Example 1 of X7 utilises oxalic acid and thus I conclude that the method exemplified involves etching. Claim 16 adds the feature 'all over the surface of the particle core'. Thus with no additional technical features added to the method and the remaining new technical feature being present in claim 1 which I have already concluded is not novel, then claims 14 and 16 are in my view also not novel.

73. Next, I must turn to claim 2 which defines the percentage of the surface area occupied by pillars. The text of X7 does not discuss the percentage coverage of nanowires and Example 1 is silent on the actual percentage surface area covered by nanowires in the generated product. The Requester states confidently that "Fig.1a of X4 shows that the nanowires occupy a percentage of the surface of the Si/Fe cores that falls within the range of 10 to 50%. The same is true of Fig. 2 of X7 (or X8 when viewed for clarity purposes)." I am afraid that I do not share the Requester's ability to make this determination (I assume that the reference to Fig. 2 was intended to refer to Fig.1 as Figure 2 shows the prior art), even looking at the clearer X8 image (reproduced below), and thus I do not feel I have the evidence to state that claim 2 is anticipated by X7/X8.

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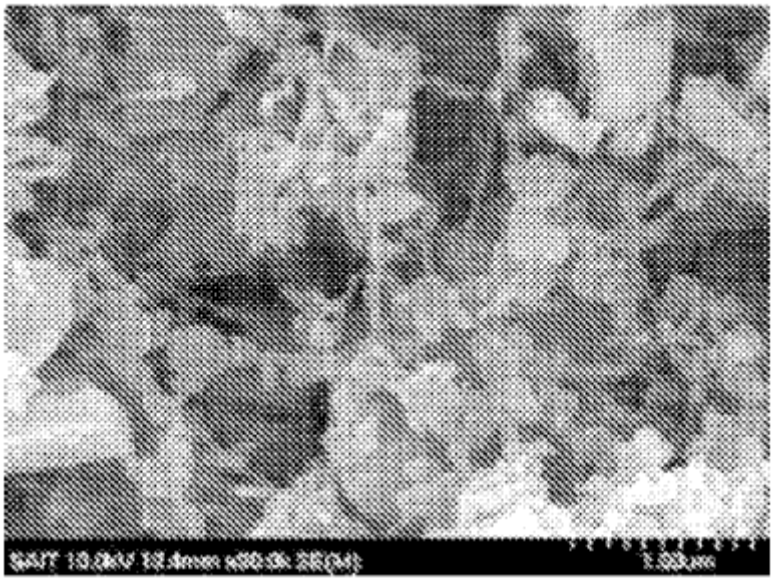

74. The Requester has not asserted that any of the remaining dependent claims lack novelty with regard to the disclosure of X7, but I believe that claims 4 and 7 are also anticipated. Claims 4 and 7 are both defined in terms of properties ('pillars arranged in a regular or irregular array' and 'compris[ing] undoped silicon, doped silicon…') which barely limit, if at all, the scope of the invention beyond that defined in claim 1, e.g. if present, silicon is either doped or undoped and if it is present in a mixture it "comprises" both. If claims 4 and 7 are not novel and nor is claim 14 then it follows that claim 17 must also not be novel.

75. Therefore I conclude that X7 is a novelty destroying document against claims 1, 4, 7, 12, 13, 14, 16 and 17.

X9, X10 and X11

76. X9 relates to what is described at paragraph 0014 of the document as *"a porous silicon (Si) powder comprising silicon particles, wherein the outermost layers of said particles are porous…The method of creating such porous powders involves treating the silicon powders making up a silicon powder with an electroless chemical etching technique known as stain-etching"*.

77. X10 page 3 lines 13-14 and lines 22-26 describes *"[p]orous silicon particles … prepared from a metallurgical grade silicon powder"* and states that *the "porous silicon particles of the present invention are unique in that each particle has a solid core surrounded by a porous silicon layer …"* and *"the porous silicon particles"* are *"useful as…electrodes in fuel cells"*. The Requester further draws my attention to the passage at page 7 lines 3-4 which describes *"[i]n etching the isolated silicon particles, any etching method known to one of ordinary skill in the art may be employed"*.

78. Finally X11 describes negative active material for lithium rechargeable batteries including porous particles having a plurality of voids therein (see paragraph

0017) where the “negative active material” is an aggregate of porous silicon particles (see paragraph 0016).

79. The Requester argues in the original request that each of the disclosures of these documents deprives claim 1 of novelty. In addition, in the observations in reply the Requester expands on the basis for this assertion stating:

> “stain etching clearly can lead to protrusions that meet Green et al.’s definition of pillars.
> Furthermore, as is well-known in the art, a porous layer contains both pores and pillars, which respective distribution depends upon the degree of porosity. Increasing the porosity enlarges the pores, thereby promoting a merging of neighbouring pores and isolation of silicon columns.”

80. The Observer maintains instead that:

> “as explained … with respect to how the claims of the Patent should be construed, we submit that a porous surface layer does not comprise [a] plurality of silicon-comprising pillars extending all over a surface of the particle core, as presently claimed. Therefore, the claims of the Patent are novel over X9, X10 and X11.
> This is supported by the enclosed Declaration of Professor Canham, who states that the stain-etching technique used in X9 would produce particles with a porous layer comprising a highly interconnected honeycomb lattice … This technique would not produce silicon particles with pillars extending from the core, as presently claimed…”

81. Before I try to reach a conclusion as to the novelty or otherwise of claims 1 and 14 with respect to documents X9-X11, I think that it would be useful to quote the well-known judgement in the Court of Appeal in *General Tire & Rubber Company v Firestone Tyre & Rubber Company Limited*, [1972] RPC 457 in relation to what is required for a novelty-destroying disclosure:

> *"If, on the other hand, the prior publication contains a direction which is capable of being carried out in a manner which would infringe the patentee's claim, but would be at least as likely to be carried out in a way which would not do so, the patentee's claim will not have been anticipated, although it may fail on the ground of obviousness. To anticipate the patentee's claim the prior publication must contain clear and unmistakable directions to do what the patentee claims to have invented ... A signpost, however clear, upon the road to the patentee’s invention will not suffice. The prior inventor must be clearly shown to have planted his flag at the precise destination before the patentee".*

82. Whilst the ‘post-infringement test’ is now complicated by the decision in the Supreme Court in *Eli Lilly v Actavis,*[5] disregarding equivalents, the principle is still sound, and this wording follows on from the statement of that test.

83. The statement from *General Tire* is helpful because I maintain it means I do not need to determine whether or not the methods utilised in X9-X11 could bring

[5] *Eli Lilly v Actavis UK Ltd & Ors* [2017] UKSC 48

about a pillared particle as defined in claim 1. All three of X9, X10 and X11 as discussed above teach porous silicon particles. The inevitable result of following the instructions in these documents would be porous silicon particles. It is clear to me that whether or not stain etching can bring about pillared particles when the porosity within a porous silicon layer is increased to the point that neighbouring pores merge resulting in the isolation of silicon columns as is suggested by the Requester, it is not the inevitable result of carrying out the methods in these documents. Therefore according to *General Tire* the Patent's claim has not been anticipated. Thus I consider claims 1 and 14 to be novel over X9, X10 and X11.

Conclusion

84. I am of the opinion that the specification of the Patent does not disclose the invention as defined in claims 1-17 clearly enough and completely enough for it to be performed by a person skilled in the art, and that claims 1, 4, 7, 12, 13, 14, 16 and 17 are not novel on the basis of document X7.

Application for review

85. Under section 74B and rule 98, the proprietor may, within three months of the date of issue of this opinion, apply to the comptroller for a review of the opinion.

Simon Grand
Examiner

NOTE

This opinion is not based on the outcome of fully litigated proceedings. Rather, it is based on whatever material the persons requesting the opinion and filing observations have chosen to put before the Office.

Annexe 1

Claims for granted patent EP 2194596 A1:

1. A particle for an electrode comprising silicon, the particle having a silicon-comprising particle core and a plurality of silicon-comprising pillars extending all over the surface of the particle core, wherein the pillars are electrochemically active.

2. A particle as claimed in claim 1 wherein the percentage of the surface areas of the particle core occupied by pillars is 10 to 50%.

3. A particle as claimed in any preceding claim wherein the pillars extend over one or more crystal faces of the particle core.

4. A particle as claimed in any preceding claim wherein the plurality of pillars are arranged in a regular or irregular array.

5. A particle as claimed in any preceding claim in which the pillars have a first dimensions in the range 0.08 to 0.70 microns, and/or a second dimension in the range 4 to 100 microns.

6. A particle as claimed in any preceding claim in which the pillars have an aspect ratio of greater than 20:1.

7. A particle as claimed in any preceding claim in which the particle and/or pillars comprise undoped silicon, doped silicon, an alloy of silicon or a silicon germanium mixture.

8. A particle as claimed in any preceding claim in which the particle and/or pillars comprise n-type or p-type silicon.

9. A particle as claimed in any preceding claim in which the particle and/or pillars have a resistivity of 0.001 to 100 Ohm cm.

10. A particle as claimed in any preceding claim wherein the purity of the silicon content is 90.00 to 99.95% by mass.

11. A particle as claimed in any preceding claim wherein the particle is metallurgical grade silicon.

12. A particle as claimed in any preceding claim having a first dimension of 10 µm to 1 mm.

13. An electrode containing particles as defined in any preceding claim as one of its active materials.

14. A method of forming a particle for an electrode, the method comprising etching a silicon-comprising particle to form a silicon-comprising particle core and a plurality of silicon-comprising pillars extending therefrom, wherein the pillars are electrochemically active.

15. A method as claimed in claim 14 wherein the etching is chemical reaction etching or galvanic exchange etching.

16. A method as claimed in claim 14 or 15 in which the pillars extend all over the surface of the particle core.

17. A method as claimed in any of claims 14 to 16 of forming particles as claimed in any of claims 2 to 12.