

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

POSCO Future M Co., Ltd.,
Petitioner,

v.

TIAX LLC,
Patent Owner.

U.S. Patent No. 9,391,317 B2
Title: Polycrystalline Metal Oxide, Methods of Manufacture Thereof, and Articles
Comprising the Same

**PETITION FOR *INTER PARTES* REVIEW
OF U.S. PATENT NO. 9,391,317 B2**

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I. INTRODUCTION

U.S. Patent No. 9,391,317 (the “’317 patent”) generally pertains to cathode materials in lithium-ion batteries. A rechargeable lithium-ion battery works by moving charged lithium ions back-and-forth between a cathode (positive electrode) and an anode (negative electrode) through an electrolyte. The cathode’s composition can affect how long the battery lasts before degrading.

Conventional cathodes tended to experience cracking after repeated cycles of recharging and draining. Made of polycrystalline material, some of the cracking occurred in unstable regions where edges of adjacent crystallites abut each other, called “grain boundaries.” The ’317 patentees proclaimed to be the first to “have discovered that lithium nickel oxides ... having cobalt enriched grain boundaries are surprisingly stabilized.” Ex_1001, 3:10-12. But this was not inventive. Others already had disclosed processes that enriched grain boundaries and increased cobalt concentration in this region to stabilize the cathode material.

Lampe-Onnerud, a patent issued several years earlier to Patent Owner, disclosed using the *same* compounds as in the ’317 patent, in the *same* wide ranges of amounts, pursuant to the *same* process, to address the *same* stability problem in cathodes. Just like in the ’317 patent, Lampe-Onnerud described enriching the material by soaking it in dissolved cobalt salts, drying/evaporating the solution, and then performing two heating steps. While Lampe-Onnerud refers to the objective as creating a “cobalt concentration gradient” instead of using the words “grain boundary

enrichment,” it is axiomatic that performing the same process yields the same result, and a POSITA would have understood that enrichment occurs in “grain boundaries.” Lest there be any doubt about the ’317 patent’s striking similarity to Lampe-Onnerud’s disclosure, Petitioner replicated a process detailed in Lampe-Onnerud and confirmed that it yields material having a greater concentration of cobalt in grain boundaries than in crystallites, exactly as that is claimed in the ’317 patent.

Park, a U.S. patent application filed and published before the ’317 patent’s effective filing date, explicitly described applying a coating along grain boundaries of a polycrystalline cathode material to improve thermal stability. Park’s technique enriched grain boundaries using lithium salt, but based on disclosures in Lampe-Onnerud, a POSITA would have been motivated to further improve cathode stability by additionally enriching the cathode’s grain boundaries with cobalt.

Takagi, an earlier-published Japanese patent application, also disclosed using cobalt to address the cracking problem in conventional cathodes. In Takagi’s process, cobalt forms at the edges/surfaces of crystallites during their formation, and as heating causes the crystallites to abut each other, their edges/surfaces become grain boundaries having increased cobalt concentration. Takagi refers to a grain boundary, where adjacent crystallites agglomerate, as a “bonding interface.” Once again, Petitioner replicated an example process in Takagi and verified that the resulting polycrystalline material has a greater concentration of cobalt in a grain boundary than in crystallites.

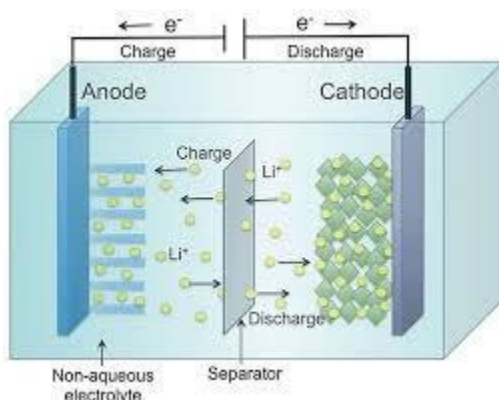
As shown below, Lampe-Onnerud and Takagi anticipate the challenged claims or render them obvious. Additionally, when Park is taken in combination with Lampe-Onnerud, the claims would have been obvious.

II. GROUNDS FOR STANDING UNDER 37 C.F.R. § 42.104(A)

Petitioners certify that the '317 patent is available for IPR, and that Petitioner is not barred or estopped from requesting an IPR of the challenged claims on the grounds identified herein.

III. TECHNOLOGY BACKGROUND

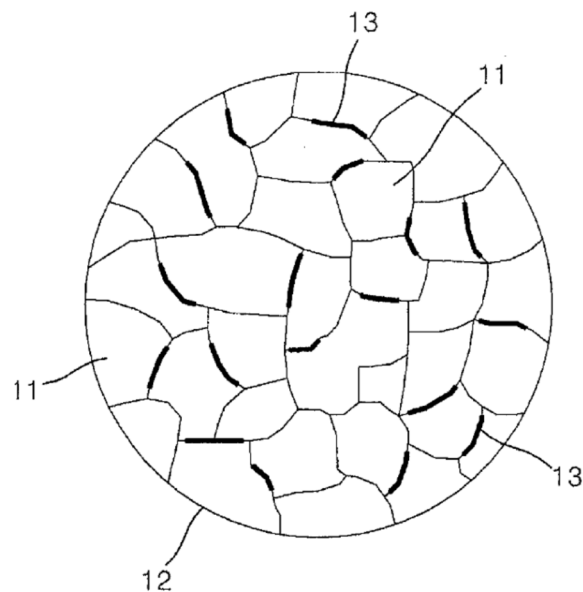
Lithium-ion batteries drain and recharge by moving positively charged lithium ions across a separator located between a cathode and an anode in an electrolyte.



Ex_1016, p.2. As the battery is charging, lithium ions move out of sites in the cathode and into sites of the anode. The reverse occurs while the battery is draining. The efficiency of this process depends on the materials that are used. Ex_1002, ¶¶26-30.

Cathodes are typically made of polycrystalline material, or polycrystals. These are solids made of small “grains,” or crystallites, of different sizes and orientations. The crystallites, referred to as “primary particles,” are “agglomerated” (clustered

together under heat) to form a polycrystalline particle, referred to as a “secondary particle.” Ex_1002, ¶31. The drawing below depicts a cross-section of a secondary particle 12 (including all contents within the circle), comprised of a jigsaw puzzle-like arrangement of primary particles 11 (each jigsaw puzzle piece), as it was understood in the art.



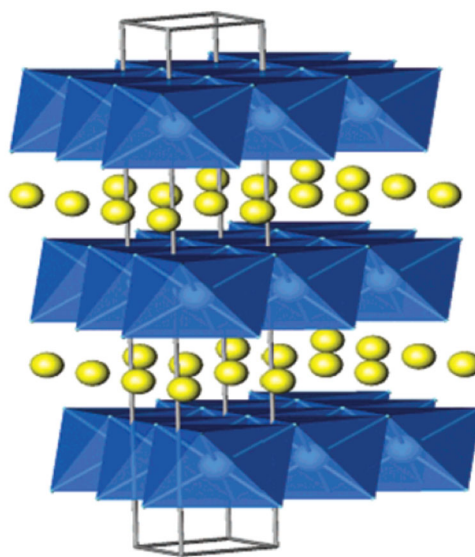
Ex_1008, FIG. 1; Ex_1002, ¶189. The edges, or surfaces, of adjacent primary particles have a shared interface, or boundary 13, called a “grain boundary.” *Id.*

Grain boundaries form naturally as “defects” in the polycrystalline material where primary particles agglomerate (are clustered) into a secondary particle. Ex_1002, ¶32. As can be seen from the figure above (a near duplicate of which appears as FIG. 1 of the '317 patent), grain boundaries are oriented randomly in various directions. Because they are the interfaces of adjacent particles, grain

boundaries are structurally the least stable area of the polycrystalline material.

Ex_1002, ¶32.

The '317 patent pertains to polycrystalline cathode materials made of lithium metal oxides. In these materials, the primary particles naturally form a structure having alternating layers.



Ex_1015, FIG 1 (showing lithium ions in yellow between blue transition metal oxide sheets). This is commonly referred to as a layered “ α -NaFeO₂ type structure.” The lithium ions move into and out of the layers as the battery recharges and drains. Because grain boundaries (where the primary particles interface) are defect regions, the structure in those areas tends to be less uniform than at the core (or “bulk”) of the primary particles. As such, some areas in a grain boundary have a layered α -NaFeO₂ type structure whereas other areas have other, less ordered cubic structures that are not layered. Ex_1002, ¶¶32, 37-44.

Early on, cathodes for lithium-ion batteries were made of lithium cobalt oxide material (LiCoO_2) because that material is stable, but it is expensive and has only moderate capacity. It then became known (still over twenty years ago) to use lithium nickel oxide material (LiNiO_2) instead, but nickel was found to be less stable than cobalt in these materials. Ex_1002, ¶¶34-35.

When a cathode made of lithium nickel oxide material is cycling (charging and draining), the movement of lithium ions into and out of the layered $\alpha\text{-NaFeO}_2$ type structure can deform the material's structure. Because nickel ions have a smaller diameter than lithium ions, they can move into sites vacated within the lithium layer, blocking lithium ions from being reinserted. This rearrangement leads to cracking, particularly when this occurs at grain boundaries, because those areas are already less stable than the highly ordered bulk crystal. As grain boundaries that are near the surface of the secondary particle crack, this can create openings through which electrolyte solution can enter the polycrystalline material, causing damage. Ex_1002, ¶¶42-43; Ex_1011, A362.

By the early 2000s, it was known to incorporate small amounts of additional transition metals with larger diameters than nickel to stabilize the cathode polycrystals. These are referred to as lithium nickel oxide-*based* materials. Ex_1002, ¶36. Cathodes doped with small percentages of cobalt, manganese, and/or aluminum

tended to be somewhat more stable than LiNiO₂ material, but still exhibited deformation and cracking. *Id.*, ¶48.

In conventional lithium nickel oxide-based materials, the additional transition metal is uniformly distributed throughout the material. *Id.*, ¶48; *see* Ex_1008; Ex_1010; Ex_1011. But since the expansion/contraction and cracking tends to be more destructive when it occurs near and at the surfaces of the primary particles, it was known to be especially advantageous to stabilize those areas. Prior to the earliest effective date for the '317 patent, it was known to use cobalt for this purpose, resulting in higher concentration of this more stable transition metal at the grain boundaries. *Id.*, ¶¶49-50.

IV. OVERVIEW OF THE '317 PATENT

A. Specification

The '317 Patent pertains to stabilizing a polycrystalline cathode material by wetting it with a dissolved cobalt salt solution and then heating in two steps. This exact approach was already in the prior art.

As the '317 Patentees acknowledge in the Background section, problems with conventional cathode materials were already well-recognized in the art: (i) lithium cobalt oxide (LiCoO₂) material was expensive and (ii) lithium nickel oxide (LiNiO₂) was cheaper but less stable. A POSITA understood that “by substituting varying amounts of other metals for some or much of the nickel in LiNiO₂, some of the capacity and cost benefits of LiNiO₂ can be obtained with improved electrochemical

cycling stability,” and it was preferable to use “only as much Co as is required.”
Ex_1001, 1:27-45.

The patentees proclaimed they “discovered that lithium nickel oxides having the layered α -NaFeO₂-type structure and having cobalt enriched grain boundaries are surprisingly stabilized.” *Id.*, 3:10-13. They then conclude that “a lower cost material is now available that provides more of the desirable properties of LiNiO₂ with less cobalt.” *Id.*, 3:26-29.

FIG. 1 illustrates a cross-section of a “secondary particle,” which comprises “a plurality of crystallites (i.e., primary particles).” *Id.*, 3:13-14. Grain boundaries (e.g., elements 41 and 42) are at the edges/surfaces of the primary particles (e.g., element 40).

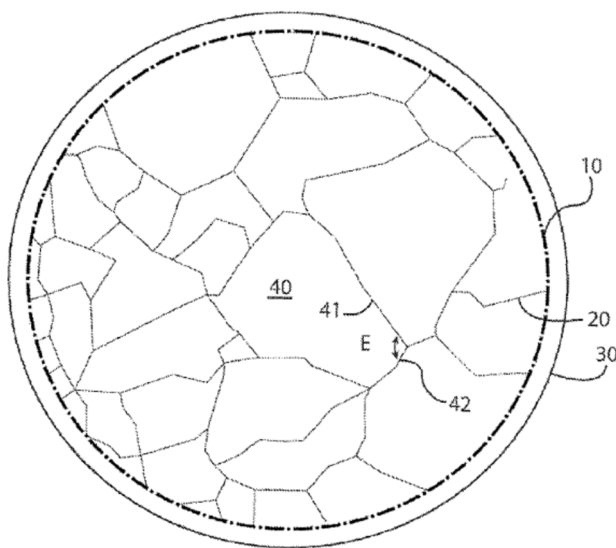


FIG. 1

Id., 6:48-50, 11:33, FIG. 1. The patent explains that a “grain boundary is between adjacent crystallites, is on a surface of the crystallite, and comprises the second

composition.” *Id.*, 6:20-21. It notes that “[t]he dimensions of the grain boundary are not particularly limited.” *Id.*, 6:66-67. Although the ’317 patent refers to FIG. 1 as illustrating an embodiment of the “invention,” the same drawing is in prior art. Ex_1008, FIG. 1.

The ’317 patent describes that the secondary particle has a “first composition” with wide ranges of lithium, nickel, cobalt, and oxygen. Ex_1001, 5:1-12. It also describes that a grain boundary “comprises the second composition,” also with wide ranges of lithium, nickel, cobalt, and oxygen. The ’317 patent provides that the same empirical formula (e.g., $\text{Li}_a\text{Ni}_{(1-x)}\text{Co}_x\text{O}_2$) can apply to the first and second compositions “with the proviso that the concentration of cobalt [(i.e., the value of “x”)] in the second composition is greater than the concentration of cobalt in the first composition.” *Id.*, 8:7-10.

The ’317 patent describes a process for enriching grain boundaries (hereinafter, the “Wetting/Heating Cobalt Enrichment Process”). A lithium nickel oxide-based polycrystalline material is mixed into an aqueous solution of dissolved lithium salts (e.g., LiNO_3) and cobalt salts (e.g., $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$). *Id.* 9:29-35. After this soaking (“wetting”), the material is heated to dry/evaporate the water, then heated in two more steps within wide ranges of temperatures and heating times:

- first, heat by “about 5 to about 20°C per minute to at about 200°C to about 500°C ... for about 0.1 to about 5 hours,” and

- second, heat by “about 5 to about 100°C per minute to at about 600°C to about 900°C ... for about 0.1 to about 10 hours.”

Id., 9:61-10:3. The '317 patent provides several examples using different amounts of lithium, nickel, and cobalt, and also varying temperatures and heating times within the disclosed wide ranges of this process.

FIG. 10 illustrates a scanning electron micrograph of a thin-cross sectional area of a secondary particle synthesized according to this process, and FIG. 13 illustrates cobalt concentrations at surfaces of primary particles in that same area:

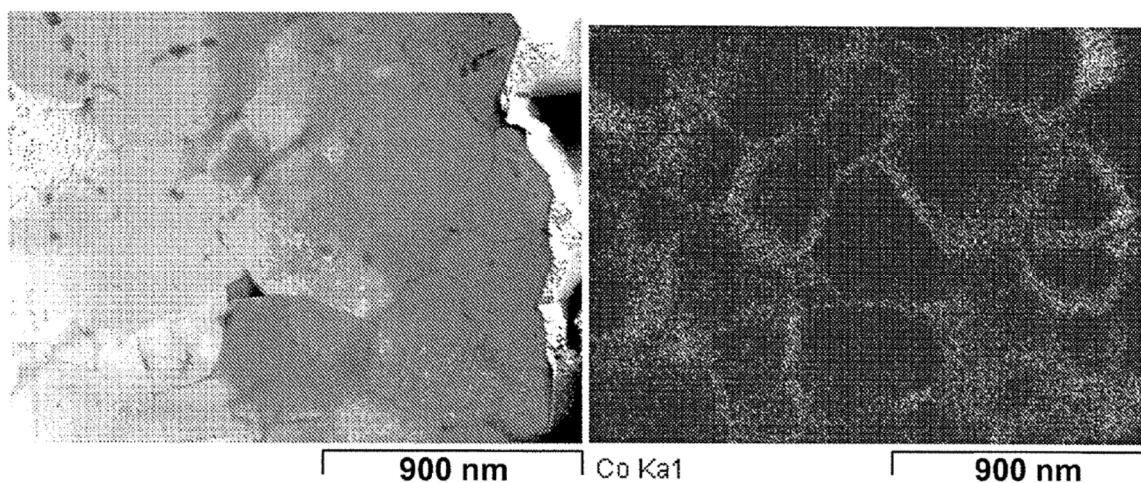


FIG. 10

FIG. 13

Id., FIGs 10 and 13. According to the patent, “[t]he image of FIG. 10 shows crystallites and grain boundaries between the crystallites,” and “FIG. 13 shows that the cobalt is concentrated at the grain boundaries.” *Id.*, 16:60-62, 17:4-5.

B. Prosecution History

The application for the '317 patent, Serial No. 14/865,805 filed September 25, 2015, claims priority to Serial No. 14/238,643 filed as PCT/US2012/048602 on

July 27, 2012 (issued as U.S. Patent No. 9,209,455), which in turn claims priority to provisional application 61/575,115, filed August 16, 2011.

In the Notice of Allowance for the parent application, the Examiner determined that while the claimed first composition was known, the prior art of record did not disclose a grain boundary between crystallites that “comprises a second composition having a concentration of cobalt greater than the first composition.” Ex_1004, pp.14-16. The application for the ’317 patent did not receive any substantive prior art rejection and was issued after filing a terminal disclaimer to overcome a non-statutory double patenting rejection. Lampe-Onnerud was identified in an Information Disclosure Statement submitted only after the Notice of Allowance was issued.

C. Representative Issued Claim

The ’317 patent includes 17 claims, of which claim 1 is independent (claim 17 is “[a] method of manufacturing the particle of claim 1”). Claim 1 recites:

1(a)	A particle, comprising: a plurality of crystallites comprising a first composition having a layered α -NaFeO ₂ -type structure and comprising
1(b)	lithium, nickel, and oxygen;
1(c)	a grain boundary between adjacent crystallites of the plurality of crystallites and ...

1(d)	... comprising a second composition having the layered α -NaFeO ₂ -type structure, a cubic structure, or a combination thereof, wherein a concentration of cobalt in the grain boundary is greater than a concentration of cobalt in the crystallites
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V. LEVEL OF SKILL IN THE ART

A person of ordinary skill in the art (“POSITA”) at the time of the ’317 Patent would have at least a master’s degree in chemistry, chemical engineering, or materials science, or related field of study, and at least three years’ experience in academia or industry researching, designing, or developing lithium-ion battery materials, which would include performing experiments to synthesize and characterize polycrystalline materials. Ex_1002, ¶78.

VI. CLAIM CONSTRUCTION

The Board gives claims their ordinary and customary meaning, or “the meaning that the term would have to a person of ordinary skill in the art in question at the time of the invention.” *Phillips v. AWH Corp.*, 415 F.3d 1303, 1312 (Fed. Cir. 2005) (en banc); 37 C.F.R. § 42.100(b). Words in a claim are given the meaning understood by a POSITA in view of the patent and file history. *Phillips*, 415 F.3d 1303, 1312-13.

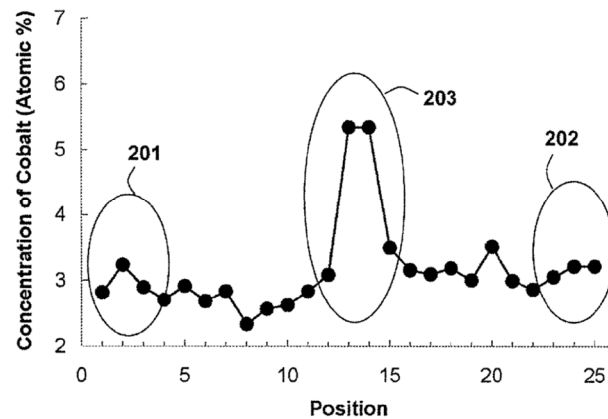
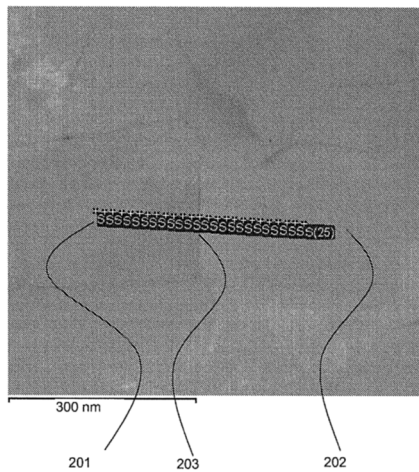
A. “grain boundary”

The term “grain boundary” is recited in claims 1-5, 11, 13 and 14, and the term appears over 120 times in the ’317 Patent specification. A POSITA would understand that, in the context of the ’317 Patent, the term refers to *a region located generally at the edges/surfaces of and between adjacent crystallites where they interface with each*

other. See Ex_1001, 6:20-26, 11:33-34, FIGs. 5, 7, 9, 10, 13; Ex_1002, ¶82. “Grain boundary” is a term of art that is well-understood by a POSITA. Ex_1002, ¶83. Petitioner’s proposed construction is supported by how the term is used consistently in the specification and also in contemporaneous patents and publications.

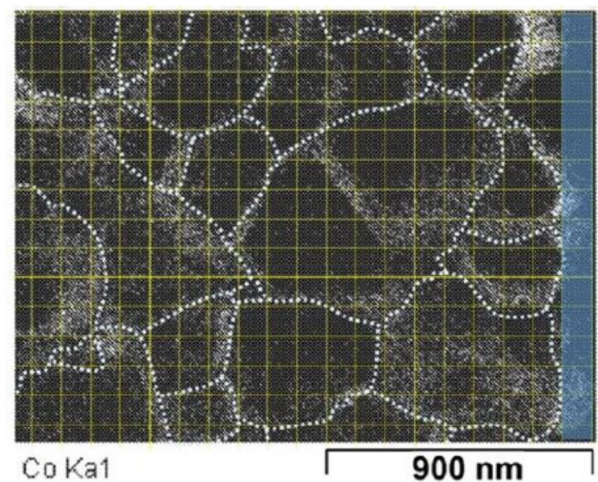
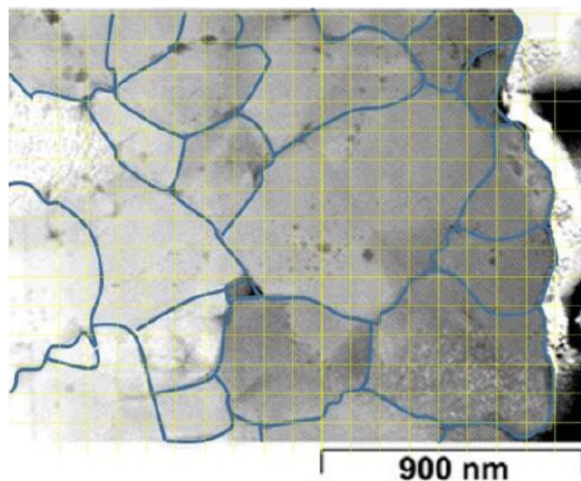
The ’317 patent specification refers to grain boundaries as being at the “edges of the crystallites,” “on a surface of the crystallite,” and “between adjacent crystallites.” Ex_1001, 6:20-25; 11:30-35. It describes the grain boundary’s dimensions as “not [being] particularly limited,” and that its length and width can generally encompass a range of “about 50 nm to 1000 nm” and its thickness can range on average from “about 1 to about 200 nanometers.” *Id.*, 6:66-7:7. In other words, a grain boundary’s thickness is generally referred to as being in a range, and the grain boundary includes the edges/surfaces of adjacent crystallites. Ex_1002, ¶¶90-92.

The ’317 patent figures illustrate that a grain boundary pertains generally to a transition region. FIG. 7 depicts how the concentration of cobalt varies at locations from the center of one crystallite to the center of an adjacent crystallite, according to a corresponding image of FIG. 5. *Id.*, 15:1-29.



Id., FIGs 5, 7. Oval 203, which corresponds to the “grain boundary” and is cross-referenced with FIG. 5, spans across locations at the edges of the adjacent primary crystals and does not have clearly defined limits. *See id.*, 15:10-16.

FIGs. 10 and 13 below are annotated with blue or dotted lines to show that the increased cobalt concentration in FIG. 13 extends beyond what can be seen as grain boundaries in FIG. 10.

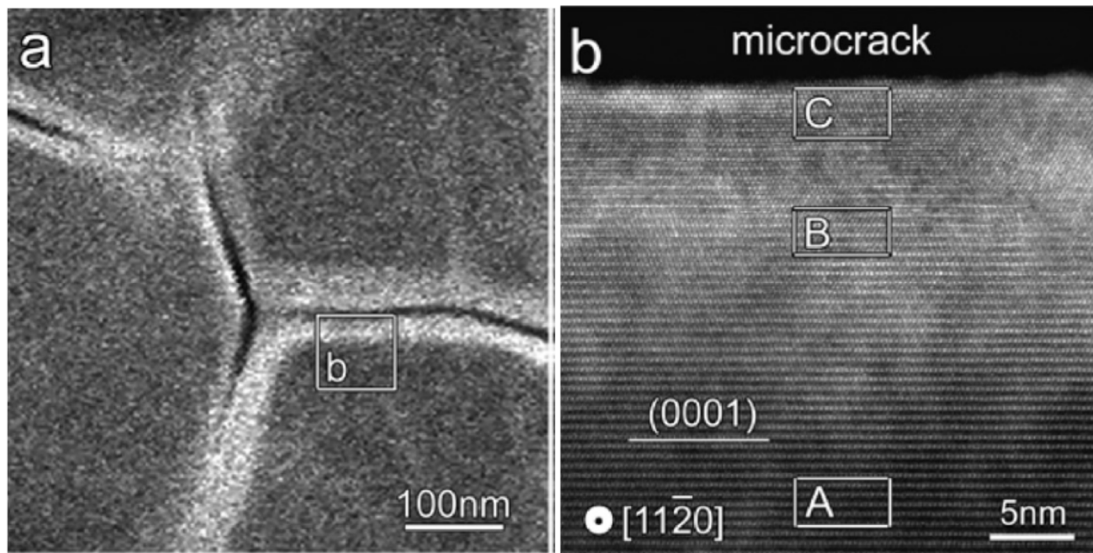


Ex_1001, FIGs 10 and 13 (annotated). The thin blue lines in FIG. 10 and dotted white lines in FIG. 13 correspond to what the patentee referred to as being grain boundaries. *Id.*, 16:60-62, 17:1-8. But the '317 patent specification also provides that “[a]n average thickness of the grain boundaries in FIG. 13 is about 100 nm.” Ex_1001, 17:5-6. According to the scale for the image, this means that the grain boundaries extend into white-shaded areas in FIG. 13, which overlap with surfaces/edges of the primary particles. Ex_1002, ¶89-91.

The '317 patentees did not invent grain boundaries, nor did they disclose any particular way to form them to have particular shapes or dimensions. Indeed, it was well understood that grain boundaries are considered defects that naturally form at interfaces of primary particles where they abut each other at their edges. Ex_1002, ¶83.

Contemporaneous literature described and showed it was known that grain boundaries are transition regions at the edges/surfaces of the primary particles where they interface. Kagei discloses that in typical lithium-ion battery cathode materials, “particles that are agglomerated so that a plurality of primary particles shares a part of their outer circumferences (grain boundaries) and are isolated from other particles are referred to as ‘secondary particles.’” Ex_1024. Zheng (shown below) discloses that in conventional lithium-ion battery cathode materials, grain boundaries are the

transition regions between interiors of primary particles and their edges. See Ex_1011, Figs 7(a), 7(b).



Id. A POSITA understood that a grain boundary is an interface between two crystals. Ex_1008, ¶22 (“[T]he term ‘grain boundary’ refers to the interface between two adjacent primary particles.”)

Consistent with the usage in the claim, the description in the '317 Patent Specification and the common understandings of the term, “grain boundary” is *a region located generally at the edges/surfaces of and between adjacent crystallites where they interface with each other.*

VII. THE CHALLENGED CLAIMS ARE UNPATENTABLE

A. Applied Prior Art References

Each asserted reference relied upon in this Petition was filed and published before July 27, 2012, the effective filing date (and also before the August 16, 2011

Provisional Application filing date)¹ and is prior art under 35 U.S.C. §§ 102(a),(b), or (e).

Prior Art References	
U.S. Pat No. 6,921,609 to Lampe-Onnerud et al., issued July 26, 2005 (“Lampe-Onnerud”).	Ex_1007
U.S. Publ. App. No. 2011/0143209 to Park et al., published June 16, 2011, filed December 1, 2010, and issued as U.S. Patent No. 8,586,247, claiming priority to Provisional No. 61/285,785 filed December 11, 2009 (“Park”)	Ex_1008
Certified Translation of Ex_1009, Japanese Published App. No. 2003-59489 A to Takagi, published February 28, 2003 (“Takagi”).	Ex_1010

B. Grounds of Unpatentability

Ground	Claim(s) Challenged	35 U.S.C. §	Reference(s)/Basis
1	1-17	102	Lampe-Onnerud
2	1-17	103	Park in view of Lampe-Onnerud
3	1-9, 11, 14-17	102	Takagi
4	10, 12-13	103	Takagi

¹This is not a proper priority claim, at least because the provisional application does not disclose steps for manufacturing the polycrystalline material, such as heating regimes, or “a second composition” in the grain boundary and structure thereof, as required by claims 1 and 17. Ex_1002, ¶¶70-74.

C. GROUND 1: CLAIMS 1-17 WERE ANTICIPATED BY LAMPE-ONNERUD

Lampe-Onnerud, titled “Gradient Cathode Material For Lithium Rechargeable Batteries,” qualifies as prior art under 35 U.S.C. §102(b).

The Background section of Lampe-Onnerud reads almost exactly like the ’317 patent. The reference similarly acknowledges that cobalt is expensive and exhibits low capacity and that lithium nickel oxide-based materials have lower cycle efficiency and are less safe due to poor structural stability. Ex_1007, 1:19-34. Lampe-Onnerud addresses a need to “minimize or overcome the above-referenced problems.” *Id.*, 1:35-37.

Like the ’317 patent, Lampe-Onnerud describes that its solution provides improved capacity, cyclability, and structural stability. *Id.*, 6:60-61. The material has the same layered α -NaFeO₂ structure in its core, with the same composition of lithium, nickel and oxygen disclosed and claimed in the ’317 patent. *See, e.g., id.*, 1:45-49, Examples.

Lampe-Onnerud discloses examples that follow the same Wetting/Heating Cobalt Enrichment Process for synthesizing the cathode material using a cobalt coating as disclosed years later in the ’317 patent. Ex_1002, ¶¶108-122, 140. Specifically, a base material (having the same “first composition” as in the ’317 patent) is wetted with dissolved lithium salt and cobalt salt, dried, and heated with ramped temperature ranges and times, within the wide ranges of the Wetting/Heating

Cobalt Enrichment Process in the '317 patent. Ex_1007, 9:62-10:12 (Example 1: ramp 5°C/minute to 450°C and hold for 1 hour, ramp 2°C/minute to 750°C and hold for 1 hour). And just like the '317 patent, the “first composition” and “second composition” have the same empirical formula. Both Lampe-Onnerud and the '317 patent disclose the exact same cobalt coating that percolates into the grain boundaries, thereby creating a “second composition” that has a greater concentration of cobalt and lower concentration of nickel.²

Lampe-Onnerud describes that the process yields a “concentration gradient” profile, without mentioning “grain boundaries.” Nonetheless, because Lampe-Onnerud teaches the same process disclosed in the '317 patent (*see* Ex_1001, 13:64-14:18) and essentially the same temperatures and heating times as in the examples of the '317 patent, the resulting secondary particle disclosed by Lampe-Onnerud necessarily has higher concentrations of cobalt in grain boundaries located near the surface of the secondary particle relative to its inner core of the crystallites as claimed by the '317 patent. Ex_1002, ¶¶108-122, 140; *see Bristol-Myers Squibb Co. v. Ben Venue Labs., Inc.*, 246 F.3d 1368, 1376 (Fed. Cir. 2001) (explaining that newly

²The Lampe-Onnerud patent and the '317 patent have the same assignee, same Power of Attorney signatory, and same prosecution counsel. Ex_1025, 211, 232, 274. They disclose the same process, even with the same typographical errors. Yet the prior art reference was not disclosed to the Examiner during examination of the '317 patent until after receiving the Notice of Allowance. *Id.*, 15-18, 161-63, 176-78. The assignee managed to secure broader claims in the '317 patent on the same cathode material made by the same process that it disclosed in Lampe-Onnerud years earlier.

discovered results of known processes are not patentable because they are inherent in the known processes). The disclosures still anticipate the '317 patent claims regardless of whether the Lampe-Onnerud inventors mentioned (or even appreciated at the time) that their process necessarily causes increased cobalt concentration in grain boundaries. *MEHL/Biophile Int'l Corp. v. Milgraum*, 192 F.3d 1362, 1365 (Fed. Cir. 1999) (“Inherency is not necessarily coterminous with the knowledge of those of ordinary skill in the art. Artisans of ordinary skill may not recognize the inherent characteristics or functioning of the prior art.”)

To further confirm that Lampe-Onnerud's secondary particles inherently include higher concentration of cobalt in a grain boundary relative to crystallites' inner cores, Petitioner's Dr. Noh replicated the process disclosed as Lampe-Onnerud's Example 1, and also replicated an example from the '317 patent. Ex_1003, ¶¶7-37. In Example 1, the base material has an “ $\text{LiNi}_{0.8}\text{Co}_{0.2}\text{O}_2$ core,” within the ranges for the “first composition” of lithium, nickel, cobalt, and oxygen disclosed in the '317 patent. Ex_1007, 9:26-10:16; Ex_1001, 12:7-15. Considering that Lampe-Onnerud and the '317 patent disclose essentially the same process, Ex_1002, ¶109, it is not surprising that Petitioner's replication of Lampe-Onnerud's Example 1 yields cross-sectional images that are extremely similar to those in the '317 patent, and that the detected cobalt concentration in the grain boundary is higher than in the crystallites.

Because the Wetting/Heating Cobalt Enrichment Process disclosed in the '317 patent and in Lampe-Onnerud is the same, the product claims (1-16) and the method claim (17) are anticipated. The images generated from Petitioner's replication of Lampe-Onnerud's Example 1 provide useful illustrations to confirm this.

1. Claim 1

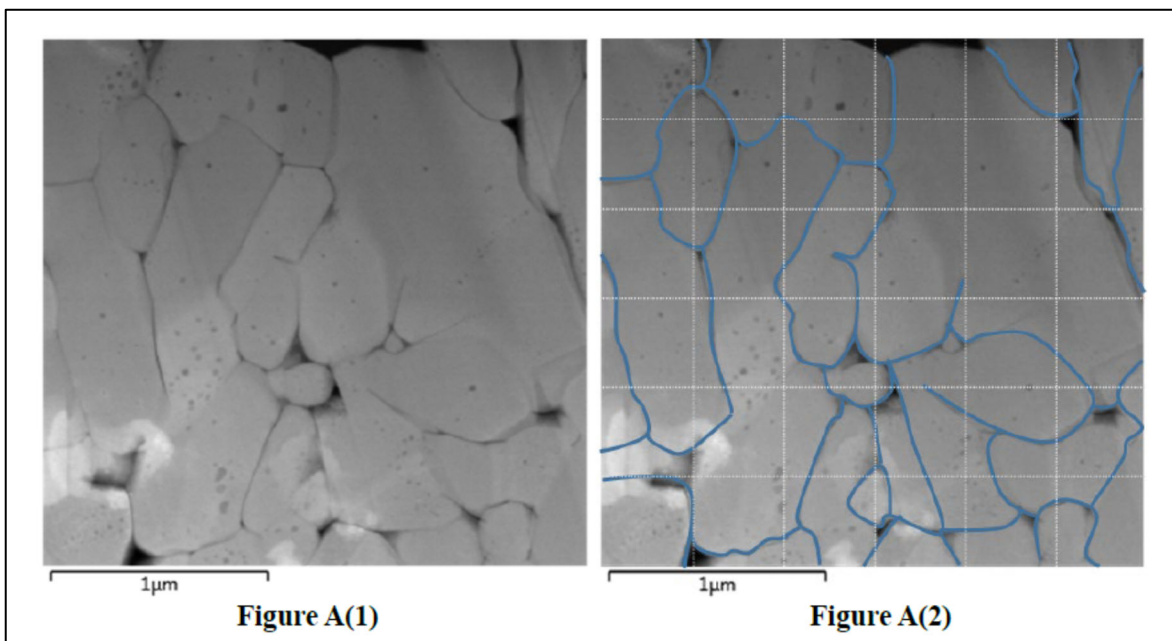
- a) **“A particle, comprising: a plurality of crystallites comprising a first composition having a layered α -NaFeO₂-type structure and comprising: lithium, nickel, and oxygen”**

Lampe-Onnerud discloses a polycrystalline particle of cathode active material. Ex_1002, ¶124; Ex_1007, 3:47-50 (“The composition of the invention is suitable for employment as the active material in cathodes.”). The material is comprised of lithium nickel oxide-based secondary particles, which Lampe-Onnerud refers to as the “crystal.”³ Ex_1007, 2:46-47 (“Li_xNiO₂ crystal structure”), 4:45-46 (“[T]he invention is a composition, such as a crystal ...”), 3:46-47 (“The crystal structure of the core ...”). Lampe-Onnerud describes that particle sizes can be 50 μ m, which confirms to a POSITA that it is referring to a secondary particle. *Id.*, 9:55-56 (“The particle size distribution ranges between 1-50 μ m after grinding.”); Ex_1002, ¶124.

³ Whether the base material is purchased (in Example 1) or synthesized (in Example 6), the material disclosed by Lampe-Onnerud includes secondary particles made of agglomerated primary particles. Ex_1002, ¶¶110-113. As further confirmation, Onnerud (having common inventors) synthesized the base material in a remarkably similar process to Example 6 of Lampe-Onnerud and provided an image showing resulting secondary particles. *Id.*; Ex_1027, 11:10-42, FIG 1.

Lampe-Onnerud discloses a “first composition” having an empirical formula ($\text{Li}_x\text{A}_{x2}\text{Ni}_{1-y1-z1}\text{Co}_{y1}\text{Q}_z\text{O}_a$) of lithium, nickel, and oxygen for primary particles in the “core.” *Id.*, 4:22-23; Ex_1002, ¶125. The reference confirms that this first composition has a layered $\alpha\text{-NaFeO}_2$ -type structure. *Id.*, 3:45-50 (“The crystal structure of the core ... is of an ‘ $\alpha\text{-NaFeO}_2$ -type.’”); Ex_1002, ¶126. It was commonly understood that such lithium nickel oxide-based cathode materials have this structure. Ex_1012, p.940 (“ LiNiO_2 , which also forms the $\alpha\text{-NaFeO}_2$ structure ...”); Ex_1013, §1.1 (LiNiO_2 atoms are arranged in a manner that leads to “a 2D layer structure, isostructural with $\alpha\text{-NaFeO}_2$ ”); Ex_1002, ¶127.

Petitioner’s replication of Example 1 of Lampe-Onnerud confirmed that performing the disclosed process necessarily yields a plurality of crystallites, which are agglomerated primary particles. Ex_1002, ¶128. The crystallites are visible in a scanning transmission electron microscope (“STEM”) image taken of a cross-section of the sample.



Ex_1003, ¶14, FIGS. A(1) (raw image), A(2) (image with traced blue lines to delineate individual crystallites); Ex_1002, ¶128. This image is very similar to FIG. 10 of the '317 patent.

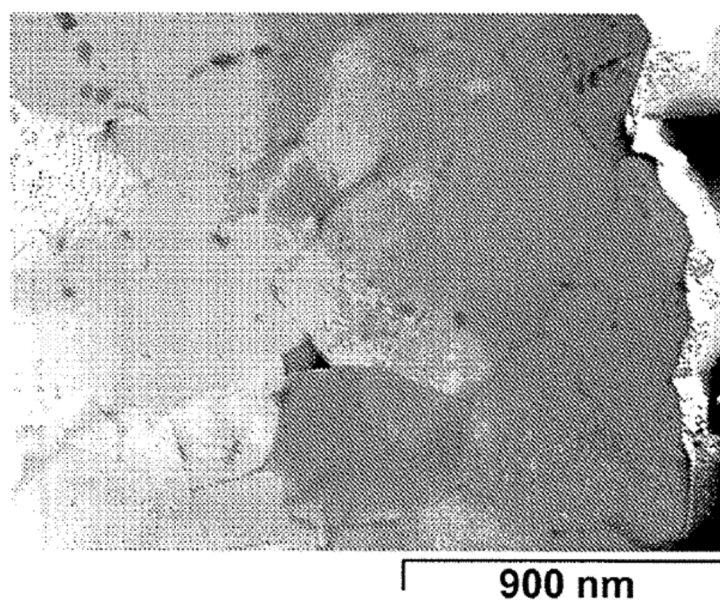
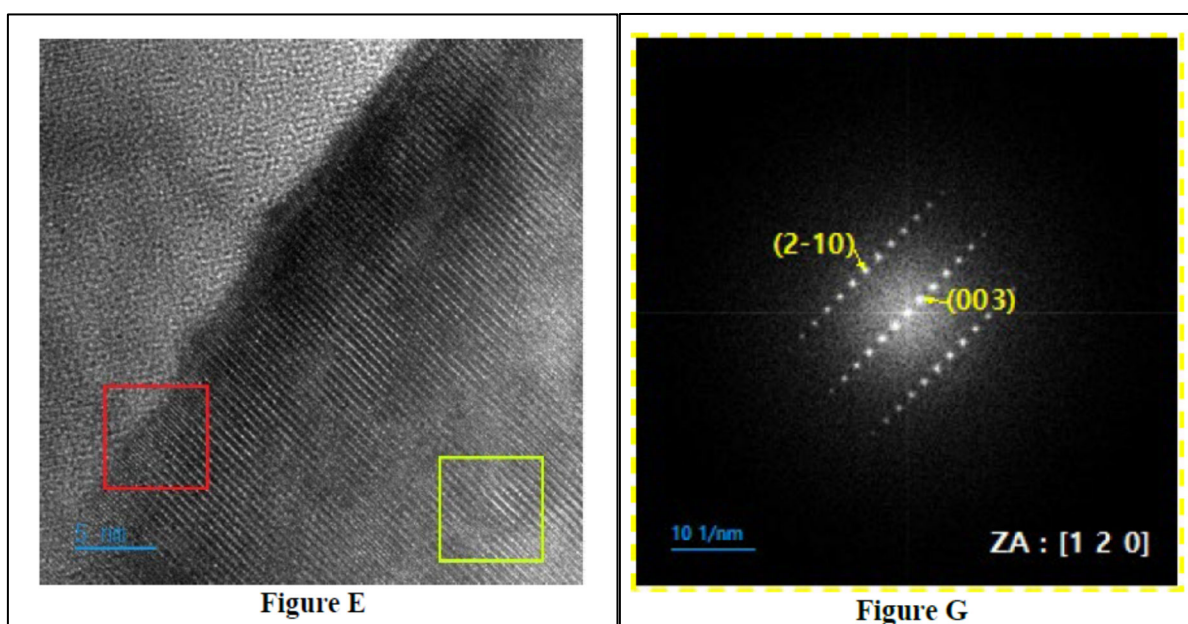


FIG. 10

Ex_1001, FIG. 10; Ex_1002, ¶131.

Petitioner's replication of Example 1 of Lampe-Onnerud also confirmed that primary particles have a layered α -NaFeO₂-type structure in the bulk. Ex_1002, ¶129. The image below at the left identifies an area (yellow box) in the “bulk” of a primary particle, away from the grain boundary (red box). On the right, using a fast fourier transform of the area in the yellow box shows a pattern of parallel dotted lines, indicating a layered α -NaFeO₂-type structure.

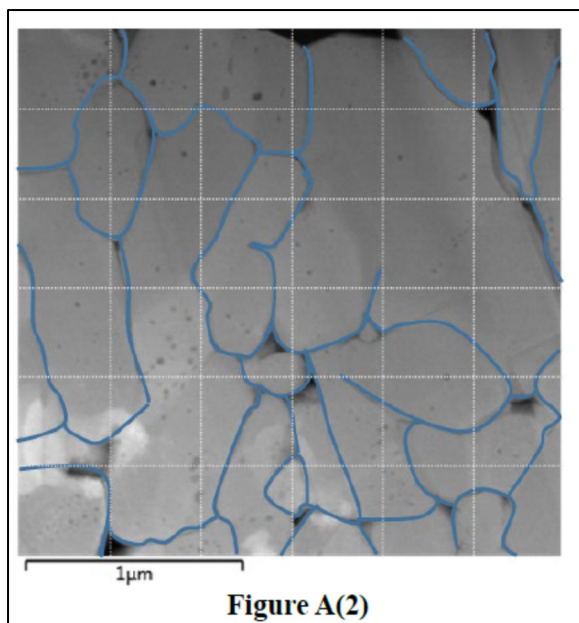


Ex_1003, ¶¶22-23, 25, FIGS. E, G; Ex_1002, ¶129.

- b) “a grain boundary between adjacent crystallites of the plurality of crystallites and comprising a second composition having the layered α -NaFeO₂-type structure, a cubic structure, or a combination thereof,”

The lithium nickel oxide-based cathode materials disclosed in Lampe-Onnerud include grain boundaries having a second composition and the claimed structure.

As discussed above in Section VII.C.1(a), performing Lampe-Onnerud's process necessarily results in a polycrystalline material having grain boundaries. Ex_1002, ¶¶108-122. Petitioner's experimentation of Lampe-Onnerud's Example 1 confirms that a cross-section of the resulting material shows a secondary particle. The traced blue lines are in grain boundaries.

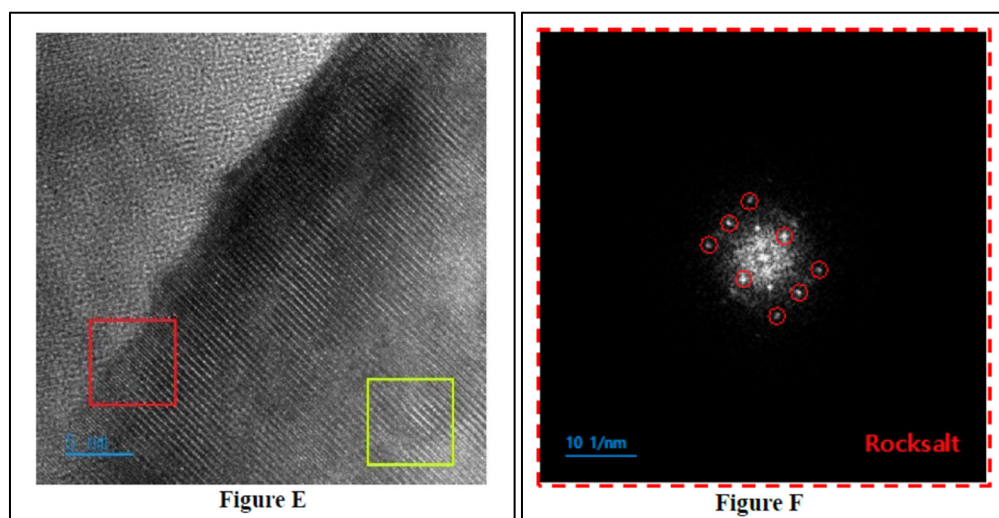


Ex_1003, Figure A(2); Ex_1001. The grain boundaries are at the surfaces of the agglomerated primary particles, where they interface with each other. Ex_1002, ¶¶81-98, *see* Section VI.A, *supra*.

As explained below in Section VII.C.1(c), Lampe-Onnerud's polycrystalline material includes grain boundaries that are a "second composition" having a greater ratio of cobalt to nickel than in the first composition. This necessarily results from Lampe-Onnerud's process of wetting a base material with a dissolved cobalt salt and

then heating to create what Lampe-Onnerud describes as a cobalt “concentration gradient.” Ex_1007, 9:44-45; Ex_1002, 102-129.

Areas in grain boundaries in Lampe-Onnerud’s polycrystalline material will have either a layered α -NaFeO₂-type structure (like the “first composition”), a cubic structure, or a combination thereof. Ex_1002, 130-136. For example, the Petitioner’s replication of Example 1 of Lampe-Onnerud shows a location in a grain boundary (red square) having a cubic (i.e., not layered) structure:



Ex_1003, ¶25, Figures E, F; Ex_1002, ¶133. This occurs because grain boundaries are defect regions “where the structure gradually changes from an ordered layer structure (α -NaFeO₂-type) to a partially ordered structure and then to a disordered rock-salt structure.” Ex_1011, Abstract; Ex_1002, ¶134-135.

Additionally, a POSITA also would have understood from Lampe-Onnerud that at least some areas of grain boundaries have a layered α -NaFeO₂-type structure. Ex_1002, ¶130-35; Ex_1011, Abstract. The reference describes that “[t]he crystal

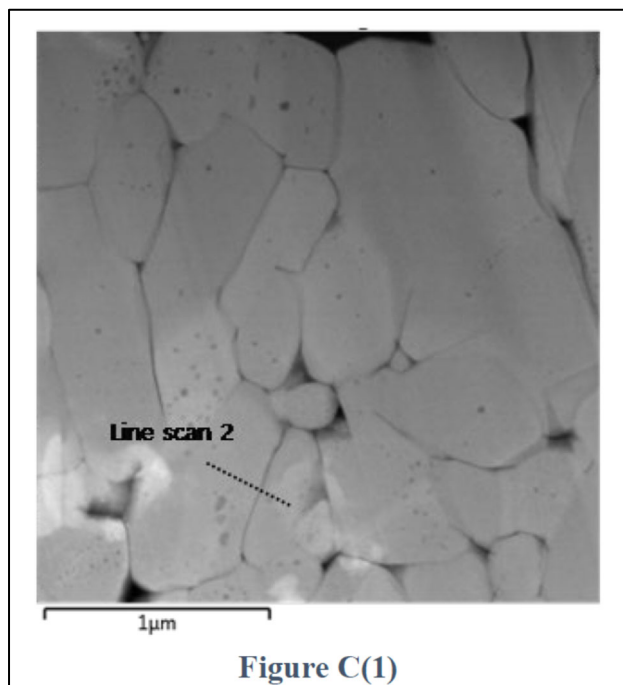
structure of the core, *and optionally of the coating*, is of an ‘ α -NaFeO₂ type.’” Ex_1007, 3:45-49 (emphasis added). Lampe-Onnerud’s coating percolates into the grain boundaries. Ex_1014, p. 2182; Ex_1002, ¶134. The coating is described as an LiCoO₂-based coating, which is known to be a layered α -NaFeO₂-type structure. *See* Ex_1012, p.940.

c) “wherein a concentration of cobalt in the grain boundary is greater than a concentration of cobalt in the crystallites.”

Lampe-Onnerud discloses a technique for creating a cobalt concentration gradient at the surface area of the secondary particle. When the dissolved cobalt salt percolates into the secondary particle and is heated, it diffuses and concentrates at grain boundaries near the secondary particle’s surface. Ex_1007, 9:44-45; Ex_1002, ¶115-119, 141. As a result, there are necessarily grain boundaries, at least near the surface of the secondary particle, that have greater concentration of cobalt than in crystallites. *Id.*, ¶121-122.

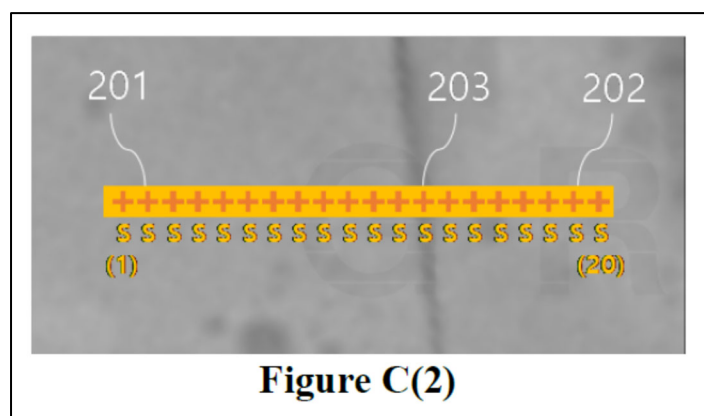
Petitioner’s experimentation of Example 1 of Lampe-Onnerud confirmed that performing Lampe-Onnerud’s process necessarily causes there to be at least an area of the material where there is higher concentration of cobalt in grain boundaries. Ex_1003, ¶20; Ex_1002, ¶¶137-141.

From Figure A(1), Petitioner performed an EDS “line scan” to examine cobalt concentration at a plurality of locations spanning from the “bulk” of one primary particle, across a grain boundary, and to the “bulk” of an adjacent primary particle.

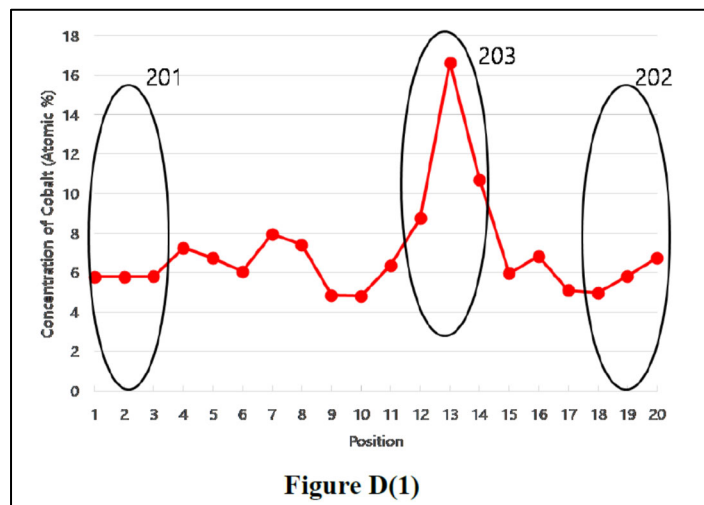


Ex_1003, ¶16, Figure C(1).

Figure C(2) is a zoomed-in image of the line scan from Figure C(1), and Figure D(1) is a plot of the cobalt concentration (atomic percentage) at 20 locations along the line scan.



Id., Figure C(2).



Id., Figure D(1). The region where the grain boundary is crossed (203) has higher cobalt concentration than the interior (“bulk”) of the adjacent primary particles, or crystallites (201, 202). Ex_1002, ¶138. Accordingly, the grain boundary is a “second composition” that has comparatively more cobalt and less nickel.⁴ *Id.*

Once again, Lampe-Onnerud yields materials with grain boundaries that are similar to those disclosed and claimed in the ’317 patent at least because they disclose the same Wetting/Heating Cobalt Enrichment Process, and both disclose examples

⁴ To confirm that Example 1 of Lampe-Onnerud yields a result that is consistent with the ’317 patent, Petitioner also replicated ’371 patent Examples 1 and 2. See Ex_1003, ¶26-37. The results were similar, both showing increased concentration of cobalt in a grain boundary as compared with crystallites. *Id.* Petitioner also replicated Examples 1 and 7 of U.S. Patent No. 7,381,496 (“Onnerud”), which discloses the same process, using similar temperatures and heating times, to further show that enriching the grain boundary with cobalt was known in prior art. Ex_1003, ¶45-48; Ex_1002, ¶141. The resulting sample from Onnerud also showed higher concentration of cobalt in grain boundary regions. Ex_1003, 49-53; Ex_1002, ¶141. Specifically, Figures Q(2) and (R) confirmed that concentration of cobalt in a grain boundary was higher than in the bulk of crystallites. Ex_1003, 51-53; Ex_1002, ¶141.

with analogous heating temperatures and times. Just like in the specification of the '317 patent, Lampe-Onnerud discloses:

- (i) dissolving a precursor lithium nickel oxide-based material into distilled water with lithium nitrate and cobalt nitrate hydrate,
- (ii) drying,
- (iii) sintering with a first temperature ramp at 5°C per minute,
- (iv) sintering with a second temperature ramp at 2°C per minute, and then
- (v) allowing to cool.

The two sintering steps are also carried out at temperatures within the ranges of the process in the '317 patent. *See* Ex_1007, 9:61-10:17; Ex_1002, ¶¶64, 107-122, 139; Ex_1001, 13:63-14:3; *In re Best*, 562 F.2d 1252, 1255 (CCPA 1977); MPEP 2112.01 (“Where the claimed and prior art products are identical or substantially identical in structure or composition, or are produced by identical or substantially identical processes, a prima facie case of either anticipation or obviousness has been established.”).

From the disclosure in Lampe-Onnerud, a POSITA would have understood that its cobalt coating helps to reduce the cracking that occurs along grain boundaries of conventional cathode materials when the battery is charging/discharging. *See*, e.g., Ex_1011, p.A359; Ex_1022, ¶141 (“Since the positive electrode active material ... is constituted by secondary particles, it seems that separation of the primary particles

(secondary particle crack) occurred due to expansion and contraction of the positive electrode active material during the charge/discharge cycles.”) A POSITA would have understood that the coating percolates into grain boundaries near the surface of a secondary particle, and with sintering, the cobalt substitutes for nickel, causing increased concentrations of cobalt that stabilizes the region. *See, e.g.,* Ex_1014; Ex_1002, ¶¶121,140 ; Ex_1007, 7:64-8:7.

Lampe-Onnerud therefore anticipates claim 1 because it discloses a cathode material with a cobalt gradient where there is a greater concentration of cobalt in grain boundaries than in bulk primary particles, which has been experimentally verified. *See SmithKline Beecham Corp. v. Apotex Corp.*, 403 F.3d 1331, 1343-44 (Fed. Cir. 2005) (finding prior art patent to a form of compound anticipated a claimed “hemihydrate form” even if the prior art did not discuss or even recognize that the compound was a “hemihydrate”).

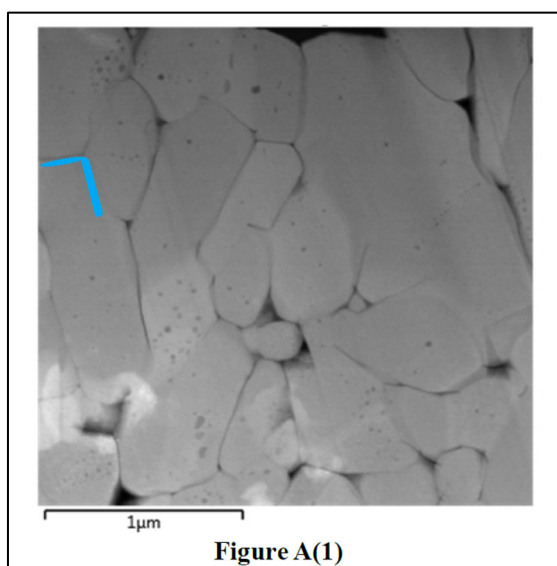
2. Claim 2

Lampe-Onnerud anticipates dependent claim 2, which further recites “wherein the grain boundary is substantially rectilinear in cross-section.”

The '317 Patent does not indicate that a process could be performed to create a specific grain boundary shape or that it has any impact on cathode performance. Ex_1002, ¶¶142-143. In any event, the polycrystalline material in Lampe-Onnerud naturally yields grain boundaries with substantially rectilinear cross-sections, i.e. that are made up of essentially straight lines when viewed in two-dimensions. Moreover,

given that Lampe-Onnerud also discloses using the same Wetting/Heating Cobalt Enrichment Process, the resulting material will be the same.

It is understood that “‘primary particles’ refers to particles that agglomerate together ... and may take various shapes such as rods or squares.” Ex_1008, ¶23; see also FIGS. 3-5; Ex_1022, FIG. 1. From the examples of Lampe-Onnerud, at least some grain boundaries formed therebetween have a substantially rectilinear shape. Ex_1002, ¶145. This is confirmed from Petitioner’s replication of Example 1 of the reference, as can be seen from the annotation of Figure A(1).



Ex_1003, FIG. A(1) (each blue line is rectilinear).

3. Claim 3

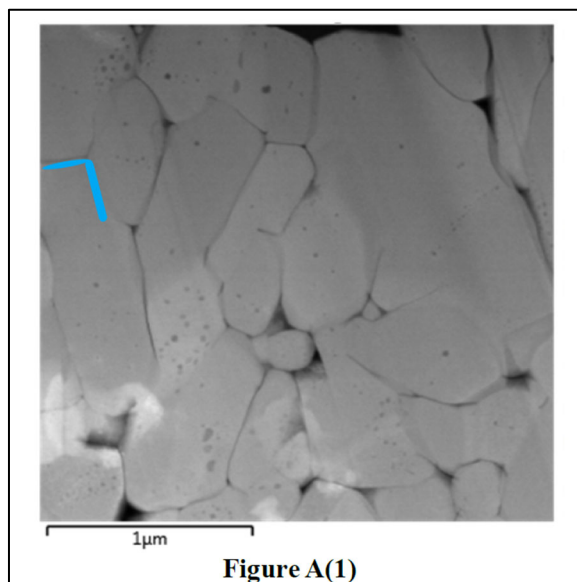
Lampe-Onnerud anticipates dependent claim 3, which further recites “wherein a direction of a surface of the grain boundary is different than a direction of a tangent of a nearest outer surface of the particle.”

It was well understood by a POSITA that a lithium nickel oxide-based polycrystalline material, such as that disclosed by Lampe-Onnerud, yields innumerable grain boundaries inside the secondary particle that form naturally and irregularly and run in various different directions. Ex_1002, ¶147-49. At least one grain boundary will be in some different direction than a tangent of the secondary particle's outer surface. *Id.*

FIG. 1 of the '317 patent illustrates a cross-sectional view of a secondary particle with grain boundaries in various random directions, nearly all of which are different than a tangent to the outer surface. Ex_1001, 6:36-39 (“[T]he surface of the grain boundary and the surface of the crystallite may have any of a variety of orientations relative to an outer surface of the secondary particle.”) Prior art also pertaining to lithium nickel oxide-based cathode material showed the same illustration. *See* Ex_1008, FIG. 1. Petitioner's experiment of Example 1 of Lampe-Onnerud yielded virtually the same image, with thin lines in various directions for grain boundaries. Ex_1003, FIGS. A(1), A(2). These lines are in different directions than a tangent of the secondary particle's outer surface. Ex_1002, ¶147-49.

4. Claim 4

Lampe-Onnerud anticipates dependent claim 4. As can be seen from the image below from Petitioner's replication of Example 1 from Lampe-Onnerud, two grain boundaries are highlighted that are on the same crystallite, intersecting at an angle according to the shape of the crystallite.



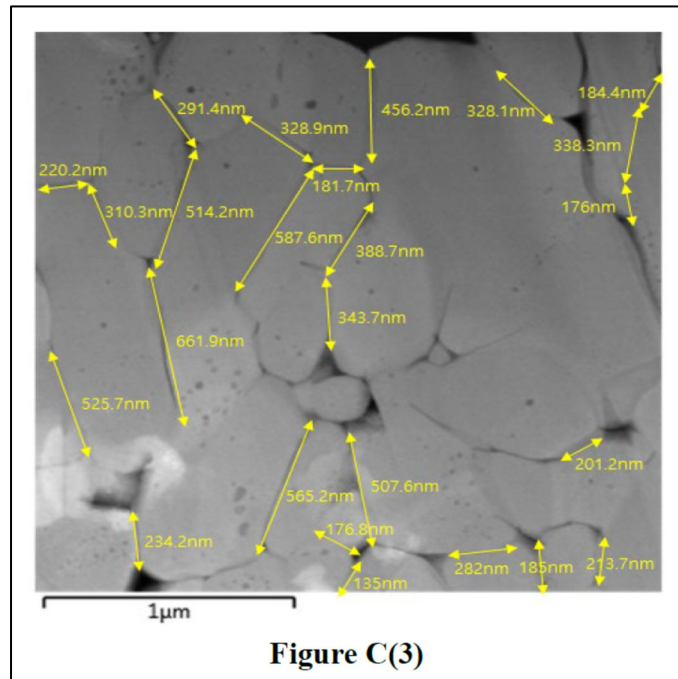
Ex_1003, Figure A(1) (annotated with blue lines intersecting at an angle). As was well understood in the art, grain boundaries form at the surfaces of primary particles where they interface adjacent primary particles, and so, from a cross-sectional view, an angle will be formed where different grain boundaries from the same primary particle intersect. Ex_1008, FIG. 1., Ex_1002, ¶¶150-154.

5. Claim 5

Dependent claim 5 further recites wide ranges for a grain boundary length (“about 50 to about 1000 nanometers”) and thickness (“about 1 to about 200 nanometers”).

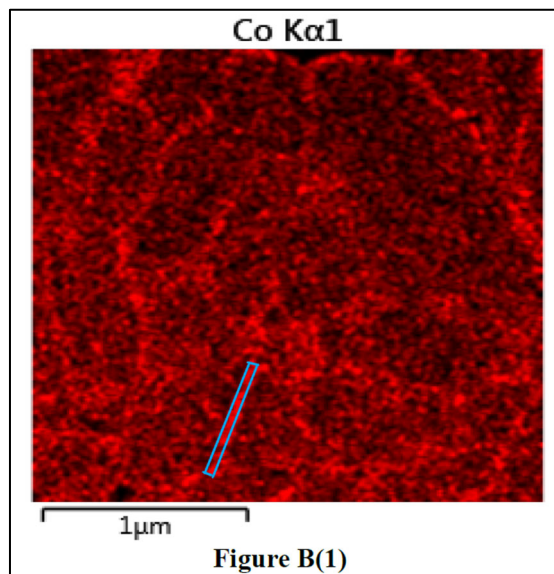
As can be seen from Figure A(1), Petitioner’s experiment of Example 1 from Lampe-Onnerud, using the embedded scale of 1 μm (1000 nanometers), includes several grain boundaries between 250 nm and 1000 nm in length. Ex_1003,

Figures A1-A2. This is confirmed from software-based measurements for many of the grain boundaries from Figure A(1) as annotated in Figure C(3).



Ex_1003, ¶19, FIG. C(3).

Also, in an STEM-EDS image from Petitioner's replication of Example 1, it can be seen that the thickness of the grain boundary annotated with a blue rectangle is between the wide ranges of 1 nm to 200 nm.



Ex_1003, Figure B(1), Ex_1002, ¶¶156-157.

The process as described in the specification of the '317 patent is practically a copy of what was disclosed in Lampe-Onnerud. Ex_1007, 9:61-10:17; Ex_1002, ¶¶64, 107-122, 139, 158; Ex_1001, 13:63-14:3. Accordingly, the same process will yield the same product with the same characteristics.

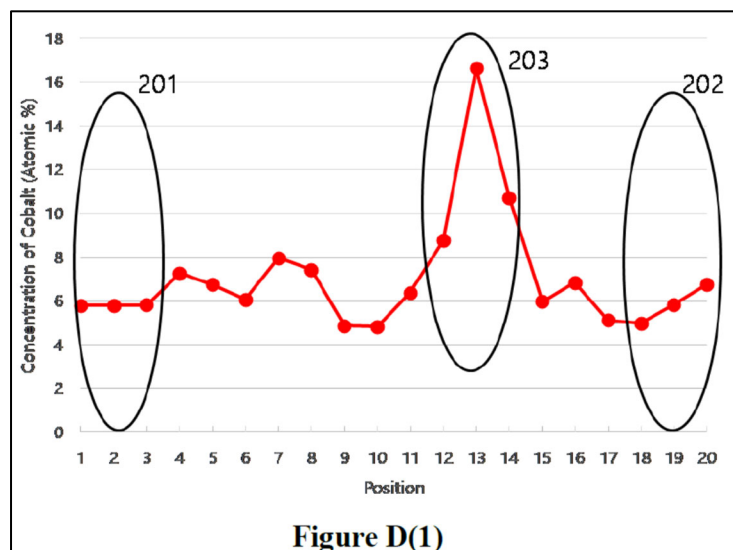
6. Claim 6

Lampe-Onnerud anticipates claim 6, which further recites that the “second composition” (in a grain boundary) has very wide ranges of possible amounts of lithium, nickel, cobalt, and oxygen.

Lampe-Onnerud discloses that the core and coating can have the empirical formula $\text{Li}_x\text{A}_{x2}\text{Ni}_{1-y1-z1}\text{Co}_{y1}\text{Q}_z\text{O}_a$ that includes lithium (Li_{x1} , where “x1” is 0.1 to 1.3), nickel ($\text{Ni}_{1-y1-z1}$ where “y1” and “z1” are between 0 and 0.2), cobalt (where “y1” is between 0 and 0.2), and oxygen (O_a , where “a” is greater than 1.5 and less than 2.1),

but arranged such that “[t]he coating has a greater ratio of cobalt to nickel than the core.” Ex_1007, 4:45-60. This results in “a gradient of diminishing nickel concentration from the core to surface of each particle.” *Id.* at 6:45-47. Accordingly, a POSITA would have understood that the coating impacts the concentrations of nickel and cobalt but will not appreciably change the lithium and oxygen. Ex_1002, ¶160. With regard to oxygen, Lampe-Onnerud discloses other empirical formulas where the oxygen amount is 2, and in each example, the amount is 2. *Id.*

Lampe-Onnerud’s disclosure is confirmed by Petitioner’s experiment of Example 1, illustrating that the concentration of cobalt in the grain boundary can be approximately 1.5x-2x of the cobalt concentration in the primary particles.



Ex_1003, Figure D(1). Accordingly, the “second composition” in Lampe-Onnerud falls exactly within the claimed ranges, as follows:

	Claim 6 ranges	Lampe-Onnerud Grain Boundary
Lithium	0.1-1.3	Li_{x1} , where “x1” is 0.1 to 1.3 (Example 1 is 1)
Nickel	0.1-1	Core is 0.6-1.0 ($\text{Ni}_{1-y1-z1}$ where “y1” and “z1” are between 0-0.2) So amount in Grain Boundary is less than 0.6-1 (up to about 2x less), which is within the range
Cobalt	0.02-0.99	Core is 0-0.2 (Co , where “y1” is between 0-0.2) (Example 1 is 0.2) So amount in Grain Boundary is greater (up to about 2x greater), which is within the range
Oxygen	1.7-2.3	O_a , where “a” is greater than 1.5 and less than 2.1 (Example 1 is 2)

Ex_1002, ¶¶161-162.

7. Claim 7

Lampe-Onnerud anticipates claim 7, which further recites that the first and second composition have an additional metal, in a very wide range from 0.01 to about 0.9 moles.

Lampe-Onnerud’s empirical formula for the core and for the coating can include any of several additional metals recited in claim 7, including Mg, V, Ti, B, Cr, Fe, and Mn. Ex_1007, 3:55-67, 4:28-60. The reference discloses that the amount of a selected additional metal can range from 0.0 to 0.5, or 0.0 to 0.2. *Id.* A POSITA would have understood that by including this metal in the precursor and in the coating, the additional metal will be present in primary particles and also grain boundaries, in

ranges according to the empirical formula, which are within the claimed ranges of 0.01 to about 0.09. Ex_1002, ¶164.

8. Claim 8

Lampe-Onnerud anticipates claim 8, which depends from claims 1 or 7 and further recites that the “first composition” contains cobalt in a range of 0 to about 0.5 mole.

Lampe-Onnerud’s empirical formula for the core can include Co in an amount that can range from 0.0 to 0.2. Ex_1007, 4:45-60. Additionally, Examples 1 and 2 include 0.2 mole of cobalt, and Examples 3, 4, and 12 have 0.1 mole of cobalt, and Examples 6, 7, and 11 have 0.08 moles of cobalt, all in the core, which includes primary particles. Ex_1002, ¶166.

9. Claim 9

Lampe-Onnerud anticipates claim 9, which depends from claim 8 and further recites that the cobalt concentration ranges from 0.0 to about 0.3. Consistent with the discussion immediately above in Section VII.C.8, the range disclosed in the reference of 0 to 0.2 is within the claimed range of 0.0 to about 0.3. Ex_1002, ¶167.

10. Claim 10

Lampe-Onnerud anticipates claim 10, which depends from claim 9 and further recites that the first composition includes nickel in a range of 0.8-1. Examples in Lampe-Onnerud are within the claimed range. For example, amounts of nickel within this range and in the core (including primary particles) are disclosed in Examples 1

and 2 ($\text{Ni}_{0.8}$), Examples 3, 6-13 ($\text{Ni}_{0.87}$), and Example 15 ($\text{Ni}_{0.90}$). Ex_1007, 9:60-16:1. Ex_1002, ¶168.

11. Claim 11

Lampe-Onnerud anticipates claim 11, which depends from claim 1 and further recites that the first and second compositions include Mn in an amount of about 0.01 to 0.6 mole.

Lampe-Onnerud's empirical formula for the core and for the coating can include Mn in an amount ranging from 0.0 to 0.2. Ex_1007, 4:35-60 (“‘Q’ is at least one element selected from the group consisting of ... manganese” and is in an amount z_1 which is “greater than about 0.0 and equal to or less than about 0.2.”) Examples 6 and 7 describe synthesis of a material having 0.05 mole of manganese in primary particles and grain boundaries. Ex_1007, 13:1-35.

A POSITA would have understood from Lampe-Onnerud that there will be a relatively uniform distribution of manganese in the primary particles and grain boundaries, and so the wide ranges of manganese disclosed in Lampe-Onnerud applies to the first composition and the second composition. Ex_1002, ¶171.

12. Claim 12

Lampe-Onnerud anticipates claim 12, which depends from claims 1 or 7 and further recites that the first composition includes nickel in a range of 0.8 to 1. This limitation is met for at least the same reasons discussed above for dependent claim 10, at Section VII.C.10. Ex_1002, ¶172.

13. Claim 13

Lampe-Onnerud anticipates claim 13, which is dependent from claim 12 and recites that the first composition comprises nickel at a concentration of 0.92 moles.

As discussed above in Section VII.C.10, Lampe-Onnerud's empirical formula provides a range of 0.6 to 1 moles of nickel in the core, and the examples are for 0.8, 0.87, and 0.90 moles of nickel. A POSITA would have understood that these ranges reflect average compositions in primary particles and that there will naturally be some variation. It was well-understood that if one were to perform the example in which a base material is synthesized with 0.90 mole of Ni (Example 15), at least some primary particles will be characterized as having slightly more or less amounts of nickel, and so if each were characterized, at least one would have an amount of 0.92. Ex_1002, ¶174.

14. Claim 14

Lampe-Onnerud anticipates claim 14, which depends from claims 1 or 7 and recites wide ranges of lithium, nickel, and cobalt in the "first composition."

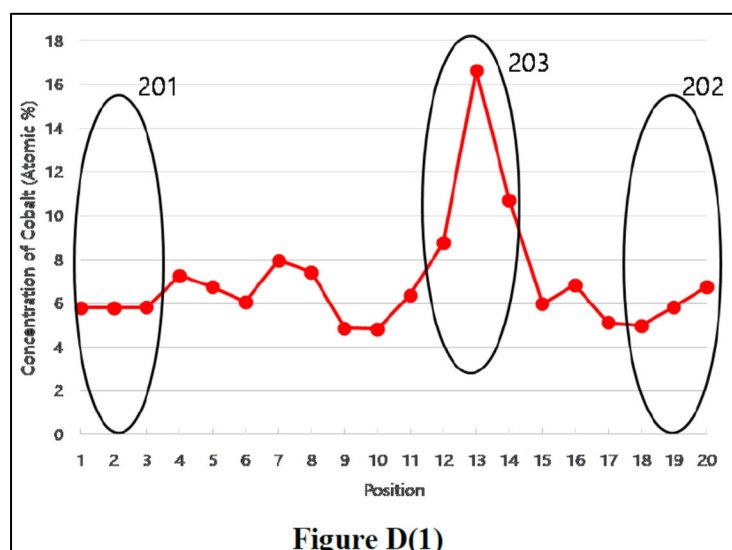
Lampe-Onnerud discloses that the core can have the empirical formula $\text{Li}_x\text{A}_{x2}\text{Ni}_{1-y1-z1}\text{Co}_{y1}\text{Q}_z\text{O}_a$ that includes lithium (Li_{x1} , where "x1" is 0.1 to 1.3), nickel ($\text{Ni}_{1-y1-z1}$ where "y1" and "z1" are between 0 and 0.2), cobalt (where "y1" is between 0 and 0.2), which is as follows:

	Claim 14 ranges	Lampe-Onnerud Core
Lithium	0.1-1.3	Li_{x1} , where “x1” is 0.1 to 1.3 (Example 1 is 1)
Nickel	0.1-1	Core is 0.6-1.0 ($\text{Ni}_{1-y1-z1}$ where “y1” and “z1” are between 0-0.2)
Cobalt	0-0.5	Core is 0-0.2 (Co, where “y1” is between 0-0.2) (Example 1 is 0.2)

See Ex_1007, 4:45-60; Ex_1002, ¶176.

15. Claim 15

Lampe-Onnerud anticipates claim 15, which is dependent from claims 1 or 7 and further recites very wide cobalt concentrations by atomic percentage. This is also inherent to performing Lampe-Onnerud’s process, as confirmed by Petitioner’s replication of Example 1, which illustrates the change by atomic percentage at various locations along a line that spans between two adjacent primary particles with a grain boundary there-between.



Ex_1003, Figure D(1). This graph illustrates cobalt concentrations in a primary particle (201 and 202) which are approximately 6-8% of the total composition of the material (within the claimed range of 0.25 to 17%) and a concentration in the grain boundary (203) of approximately 10-18% of the total composition (within the claimed range of 0.5 to 32%). Ex_1003, ¶¶20-21; Ex_1002, ¶178.

16. Claim 16

Lampe-Onnerud anticipates claim 16, which depends from claim 1 and further recites a layer on the surface of the particle. Lampe-Onnerud discloses applying a LiCoO_2 -based coating, which is an oxide, as recited in the claim. The coating also can include Al, B, or Zr, as claimed. Ex_1007, 2:57-67 (“... the coating on the core has ... at least one element selected from the group consisting of ... boron, aluminum ... and zirconium.”). Ex_1002, ¶179.

17. Claim 17

Lampe-Onnerud anticipates claim 17, which recites a method of manufacturing the particle of claim 1.

a) “.... combining a lithium compound, a cobalt compound, and a nickel compound to form a mixture;”

Example 1 discloses steps for synthesizing the claimed material, including mixing quantities of a lithium compound, cobalt compound, and nickel compound as a mixture. In particular, Example 1 discloses mixing LiNO_3 (lithium compound),

$\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (cobalt compound), and $\text{LiNi}_{0.6}\text{Co}_{0.2}\text{O}_2$ (nickel compound) in an aqueous solution. Ex_1007, 9:64-66.

- b) heating the mixture at about 30 to about 200° C. to form a dried mixture;”**

Lampe-Onnerud discloses that after the compounds are mixed as described above, they are then heated on a hot plate while stirring to evaporate the excess water. A POSITA would have understood that the “hot plate” for drying the mixture would be in a temperature range of about 30° to about 200° C. Ex_1007, 9:66-67.

- c) “heat treating the dried mixture at about 200 to about 500 °C. for about 0.1 to about 5 hours;”**

Continuing with Example 1, Lampe-Onnerud discloses that after evaporation, the dried mixture is sintered in a heat ramp at 5°C/minute to 450° C and held for 1 hour. Ex_1007, 10:2-3. This is within the claimed temperature range and timing.

- d) “heat treating at 600 to about 900 °C. for about 0.1 to about 10 hours to manufacture the particle”**

Example 1 also discloses another heating step, this time ramping by 2°C/minute to 750°C and holding for 1 hour, which is also within the claimed temperature range and timing. Ex_1007, 10:4.

- e) “wherein the particle comprises**

Claim 17 then recites the product of the process, which repeats the limitations for the particle in claim 1. In Petitioner’s replication of Example 1, the coated material was synthesized based on the mixing processes disclosed, and temperatures/timing

within the claimed range. The limitations regarding the material are therefore anticipated at least for the reasons set forth in the discussion at Sections VII.C.1.a-c (regarding claim 1). Ex_1002, ¶¶181-185.

D. GROUND 2: CLAIMS 1-17 ALSO WOULD HAVE BEEN OBVIOUS OVER PARK IN VIEW OF LAMPE-ONNERUD

Prior to the '317 patent, it was known to enrich the grain boundaries of a lithium nickel oxide-based cathode material, and that this can improve the material's stability. This was described in Park, titled "Positive Electrode Active Material for Lithium Battery and Lithium Battery Using the Same," published on June 16, 2011 as US 2011/0143209, from an application filed December 1, 2010 and a Provisional application filed December 11, 2009.⁵ Park qualifies as prior art to the '317 patent under 35 U.S.C. §§ 102(a), (b), or (e).

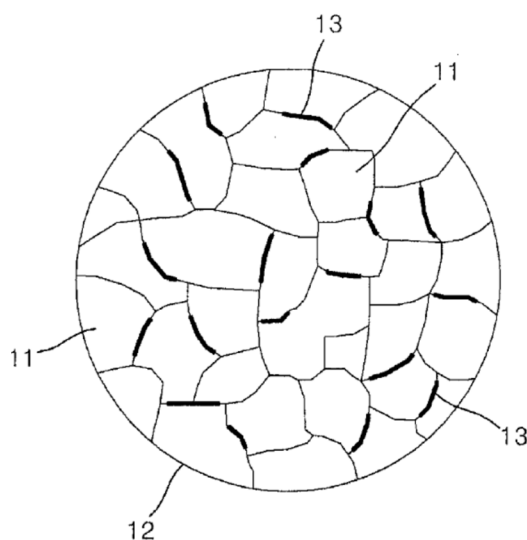
Just like in the '317 patent and Lampe-Onnerud, Park acknowledged that using lithium cobalt oxide as a cathode material is expensive and also that lithium nickel oxide was not an adequate alternative because it created stability issues and other problems. Ex_1008, ¶¶7-8. Park's solution is to enrich the grain boundaries in the material with a lithium-based material to improve its stability and capacity, through

⁵Park's provisional application has the same disclosure as in the published application (with the exception of patent claims) and so it is sufficiently supported. Ex_1002, ¶187.

creation of a buffer-like zone at the edges of the primary particles to protect the lithium nickel oxide core from structural collapse. *Id.*, ¶35, 78.

FIG. 1 of Park illustrates a secondary particle (element 12) having a plurality of primary particles (element 11) of various shapes and orientations, just like in FIG. 1 of the '317 patent.

FIG. 1



Ex_1008, FIG. 1. Consistent with what was known in the art, Park notes that “‘primary particles’ refers to particles that agglomerate together to form a secondary particle, and may take various shapes such as rods or squares.” *Id.*, ¶23. The lines at the edges of adjacent primary particles are grain boundaries. Along these grain boundaries at the surface of the secondary particle, there is an amorphous material such as a lithium salt (element 13, in darker lines) for stabilizing the material. *Id.*,

¶29. Park specifically notes that “[t]he amorphous material may be a lithium salt, but is not limited thereto.” *Id.*, ¶24.

Park discloses that the grain boundaries are enriched by mixing/stirring a lithium composite oxide with a lithium salt and then heat treating such that the resulting material “includes an agglomeration of at least two primary particles” with “amorphous material” among the grain boundaries. *Id.*, ¶12. Park provides examples of preparing the cathode material at various temperatures and then verifying using SEM that it formed secondary particles with a lithium salt in an “amorphous layer *formed along a grain boundary* of adjacent primary particles.” *Id.*, ¶64 (emphasis added). Additionally, Park confirmed that cathode materials having enriched grain boundaries emitted less heat and had improved thermal stability, thereby accomplishing the objective. *Id.*, ¶78.

While Park teaches that the “amorphous material may be a lithium salt,” it notes that other materials can be used as well. *Id.*, 24. As previously described, Lampe-Onnerud discloses diffusing a cobalt salt-based coating into the cathode active material to create a cobalt concentration gradient that improves the capacity, cyclability, structural integrity, and overall safety of the cathode active material. *See* Section VII.C.1.c, *supra*; Ex_1007, 6:53-8:7 and Example 1; Ex_1002, ¶141; Ex_1003, ¶20 and Figures C2, D1.

In combination with Lampe-Onnerud, it would have been obvious to a POSITA to modify Park by further enriching the grain boundaries with Lampe-Onnerud's cobalt salt-based coating—in addition to Park's amorphous lithium salt—to further improve the “capacity, cyclability, and safety” of the cathode material “throughout the life of the battery.” Ex_1007, 2:16-28, 6:53-7:30; Ex_1002, ¶191; and Example 1. A POSITA would have understood that additionally increasing cobalt concentration would provide even greater structural stability to the grain boundaries of the cathode material than by just forming a lithium-based amorphous material in that region. Ex_1007, 2:16-28, 6:53-7:30

1. Claim 1

- a) “A particle, comprising: a plurality of crystallites comprising a first composition having a layered α -NaFeO₂-type structure and comprising: lithium, nickel, and oxygen”**

Park discloses a lithium nickel oxide-based material according to the empirical formula $\text{Li}_a(\text{Ni}_x\text{Co}_y\text{Me}_z)_{2-a}\text{O}_2$ with nearly the same ranges of lithium, nickel, cobalt, and oxygen (e.g., $\text{LiNi}_{0.6}\text{CO}_{0.2}\text{Mn}_{0.2}\text{O}_2$) that are disclosed in the '317 patent and in Lampe-Onnerud. Ex_1008, ¶28. Park's cathode material is comprised of a plurality of agglomerated primary particles (or crystallites). Ex_1008, ¶21. Additionally, it was known to a POSITA that lithium nickel oxide-based particles have a layered α -NaFeO₂-type structure. Ex_1012, p.940; Ex_1013, p.691; Ex_1002, ¶193.

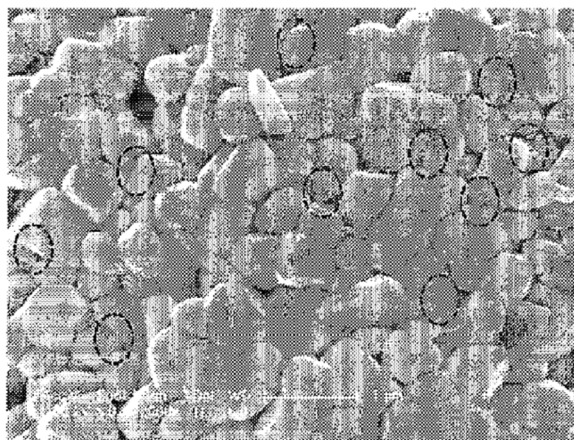
Lampe-Onnerud discloses a polycrystalline lithium nickel oxide-based core material, which has a layered α -NaFeO₂-type structure. *See* Section VII.C.1.a, *supra*.

Thus, both references correspond to this limitation.

- b) **“a grain boundary between adjacent crystallites of the plurality of crystallites and comprising a second composition having the layered α -NaFeO₂-type structure, a cubic structure, or a combination thereof,”**

Park also discloses a cathode material having grain boundaries located at the edges of adjacent primary particles, as illustrated in FIG. 1. Ex_1008, FIG. 1, ¶29. FIG. 3 of Park illustrates a scanning electron microscope (SEM) image of a cathode material prepared according to Preparation Example 1 of Park, which has “a Li₂SO₄ amorphous layer formed along a grain boundary of adjacent primary particles.” Ex_1008, FIG. 3, ¶64.

FIG. 3



Id., FIG. 3. The ovals in FIG. 3 indicate locations of grain boundaries having an amorphous layer of lithium salt. *Id.*, ¶74. The amorphous layer is part of a “second

composition” in the grain boundary distinct from the “first composition” in primary particles. Ex_1002, ¶195.

Grain boundaries in Park have a layered α -NaFeO₂-type structure, cubic structure, or a combination of both. As described in Ground 1, claim 1, it was known that lithium nickel oxide-based materials have a layered α -NaFeO₂-type structure and transition to a more disordered structure including other, similar crystal lattices at the edges/grain boundaries of the primary particles as a result of the sintering process. Ex_1011, Abstract; Ex_1002, ¶196. Accordingly, Park’s grain boundaries similarly are a second composition having some areas with a layered α -NaFeO₂-type structure and other areas having a more disordered cubic structure. By mixing the cobalt salt along with the lithium salt disclosed in Park, the grain boundaries will have a different concentration of cobalt than in the primary particles, and so the grain boundaries are a different composition. Section VII.C.1.b, *supra*, Ex_1002, ¶¶134-136.

c) “wherein a concentration of cobalt in the grain boundary is greater than a concentration of cobalt in the crystallites.”

Park discloses enriching grain boundaries to improve their stability and other properties desirable for cathode material. Park achieves this by dry mixing a lithium salt with a lithium composite oxide and then heating, to form an amorphous layer of lithium salt along the grain boundaries. While Park enriches grain boundaries, it does not disclose using cobalt. Ex_1008, ¶24. However, Park notes that the amorphous material need not be limited to lithium salt. *Id.*

Lampe-Onnerud discloses wetting a base (or core) material of lithium nickel oxide with a dissolved solution of a lithium salt (lithium nitrate) and cobalt salt. Ex_1007, Examples, Ex_1002, ¶¶107-122, 199. Like in Park, the stated purpose for Lampe-Onnerud's coating is for stabilizing the material to improve the capacity, cyclability, structural integrity and overall safety of the cathode material. *See* Section VII.C.1.c, *supra*; Ex_1007, 2:16-28, 6:53-8:7, and Example 1; Ex_1002, ¶141; Ex_1003, ¶20 and Figures C2, D1. Lampe-Onnerud describes that this process stabilizes the structure by substituting out less-stable nickel for cobalt, which occurs when the cobalt coating percolates into the material at the edges of the primary particles near the surface of the secondary particle. *Id.*, 7:64-8:7; Ex_1002, ¶56.

It would have been obvious to a POSITA to modify (or simply augment) Park to mix a cobalt salt in addition to lithium salt when forming the coating for enriching the grain boundary. Using a cobalt salt, as disclosed in Lampe-Onnerud, would additionally enrich the grain boundary with cobalt to further improve the “capacity, cyclability, and safety” of Park's cathode active material and to “enable the material to stay intact [i.e., improve the structural integrity] throughout the life of the battery.” Ex_1007, 2:16-28, 6:53-7:30, and Example 1; Ex_1002, ¶¶190-192, 200-204.

The benefit of adding lithium at the edges of the primary particle (“along the grain boundary”) as described in Park serves to effectively create a buffer-like zone to protect the crystal structure in primary particles while still providing lithium ions for

the charging/discharging process. A POSITA would have been motivated to add cobalt to this coating because it was known to be stabilizing and would provide a better protective layer than the lithium amorphous material alone. Park and Lampe-Onnerud similarly acknowledged that while cobalt is expensive when used as the primary metal, its stabilizing properties are still beneficial when used in limited amounts. Ex_1002, ¶101, 190, 200-04.

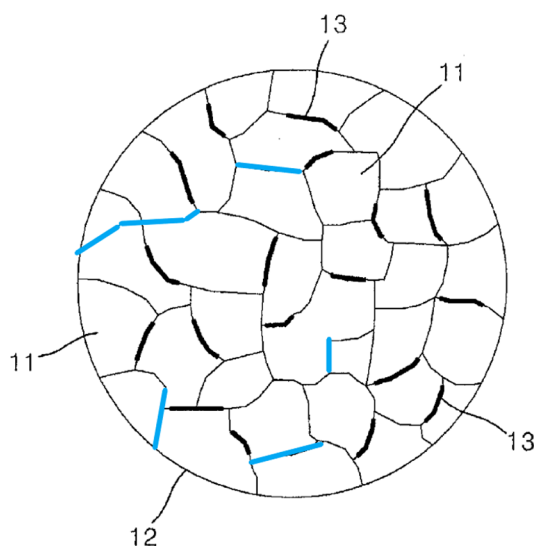
A POSITA would have understood that Park's objective to protect primary particles at the grain boundary would be easily improved by also using cobalt to create a gradient layered metal oxide in the grain boundary and at the surfaces of the primary particles which would provide even greater structural stability to the cathode material. Ex_1007, 2:16-28, 6:53-7:30, and Example 1. A POSITA would have been motivated to combine materials and would appreciate that Park and Lampe-Onnerud are seeking to address the same problem with solutions that are complementary. It also would have been obvious to a POSITA from Lampe-Onnerud to improve upon Park's concept by applying the coating using the wetting and subsequent evaporation/sintering technique in Lampe-Onnerud to ensure that the coating percolates into the surface of the secondary particle, to prevent cracking at areas where the electrolyte can seep into the material. Park also noted that it is beneficial to enrich grain boundaries "at the surface of the secondary particle 12." Ex_1008, ¶29.

Park and Lampe-Onnerud are directed to solving the same problem in a similar manner. *See* Ex_1008, ¶¶7-8, 29; Ex_1007, 4:55-57, 6:60-63, 7:6-8. A POSITA that was working with Park to improve lithium nickel oxide-based cathode material by enriching grain boundaries would have been motivated to use an additional compound to include along with the lithium salt, as Park invites the reader to do. Ex_1008, ¶24. Accordingly, Park in view of Lampe-Onnerud would have an expected result, a particle in which the concentration of cobalt in the grain boundaries is greater than its concentration in the crystallites. Ex_1002, ¶204.

2. Claim 2

Park discloses claim 2, which further recites “wherein the grain boundary is substantially rectilinear in cross-section.” As depicted in the cross-section view of FIG. 1, there are grain boundaries that are rectilinear (examples traced in blue).

FIG. 1



Ex_1008, FIG. 1 (annotated), Ex_1002, ¶189-205. Park discloses that primary particles can be shaped as squares, so the cross-sections of grain boundaries therebetween will appear rectilinear (as lines). Ex_1008, ¶23; Ex_1002, ¶189, 205.

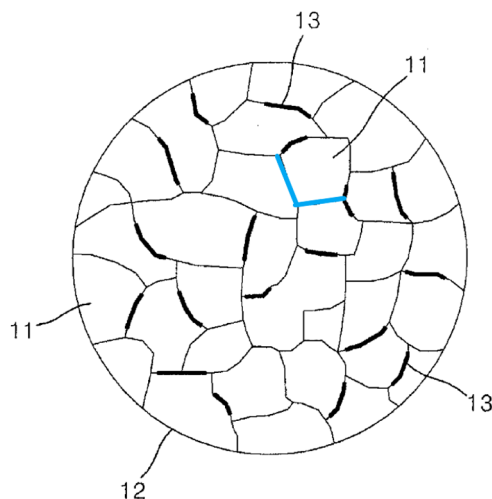
3. Claim 3

Park discloses claim 3, which further recites “wherein a direction of a surface of the grain boundary is different than a direction of a tangent ... the particle.” FIG. 1 of Park is substantially the same as FIG. 1 of the '317 patent, which is relied upon as support for claim 3. Both show a horizontal cross-sectional view of a secondary particle with grain boundaries in various random directions, nearly all of which are different than a tangent at the outer surface. Most of these lines are in different directions than a tangent of the secondary particle's outer surface. Ex_1002, ¶206.

4. Claim 4

Park also discloses claim 4. Referring again to FIG. 1 of Park and FIG. 1 of the '317 patent, there are instances where two grain boundaries are on the same crystallite and intersect at an angle according to the shape of the crystallite.

FIG. 1



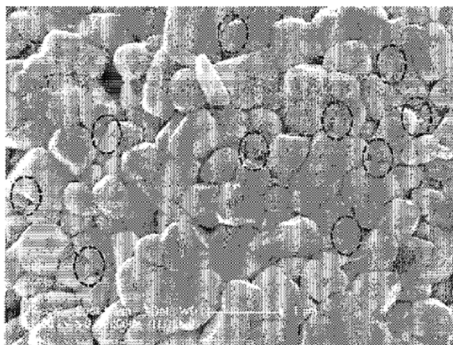
Ex_1008, FIG. 1 (annotated); Ex_1002, ¶207.

5. Claim 5

Claim 5 further recites wide ranges for a grain boundary length (“about 50 to about 1000 nanometers”) and thickness (“about 1 to about 200 nanometers”). Park discloses that the primary particles can have an average diameter of 350 nm. Ex_1008 ¶75, Table 1. Ex_1002, ¶208. Since the average grain boundary length is coincident with or less than a primary particle’s diameter, Park discloses grain boundary lengths within the wide range of the claim.

Regarding grain boundary width, FIG. 3 illustrates that grain boundaries are at intersections of the primary particles and their thickness is generally somewhat less than 5% of primary particle width, so the grain boundary thickness is within the claimed range.

FIG. 3



Ex_1008, FIG. 3; Ex_1002, ¶209.

Moreover, from Petitioner's replication of Example 1 in Lampe-Onnerud, it can be seen that the grain boundary thicknesses are approximately 100nm thick, which is within the range. Ex_1002, ¶157, citing Ex_1003, ¶15, Figure B(1) (annotated). *See* Section VII.C.1.c5. The combination of Park with Lampe-Onnerud would include a similar cobalt coating, and so grain boundary thickness would be commensurate. Ex_1002, ¶209.

6. Claim 6

Park also discloses claim 6, which further recites that the "second composition" (in a grain boundary) has very wide ranges of possible amounts of lithium, nickel, cobalt, and oxygen.

Park discloses an empirical formula of $\text{Li}_a(\text{Ni}_x\text{Co}_y\text{Me}_z)_{2-a}\text{O}_2$, where $0.5 \leq x \leq 1$, $0 \leq y \leq 0.5$, $0 < z \leq 0.5$, $0.90 < a \leq 1.15$. Ex_1008, ¶28. This is within the ranges cited in the claim. A POSITA would have known that the enrichment with lithium salt will

provide a coating in the grain boundary but will not disturb the ratios of lithium, nickel, cobalt, and oxygen. Ex_1002, ¶211.

In combination with Lampe-Onnerud, a coating with cobalt salt along with lithium salt will increase the relative concentration of cobalt and decrease the relative concentration of nickel in the region (with the amount of lithium and oxygen remaining the same), as Lampe-Onnerud discloses, but they will remain in quantities of the wide ranges that are claimed, or else that would have been obvious as part of the normal process of optimizing for producing a material with further improved stability. *Id.*; Section VII.C.6, *supra*.

7. Claim 7

Park discloses claim 7, which further recites that the first and second composition have an additional metal, in a very wide range from 0.01 to about 0.9 moles. In the empirical formula $\text{Li}_a(\text{Ni}_x\text{Co}_y\text{Me}_z)_{2-a}\text{O}_2$, “Me” can include titanium (Ti) or manganese (Mn), as also recited in claim 7. Ex_1008, ¶28. The amounts of the selected transitional metal can range can be, for example, 0.3 moles (when $z=0.3$ and $a=1$), which is squarely in the claimed range. Ex_1002, ¶212.

8. Claim 8

Park also discloses claim 8, which further recites cobalt in a range of 0 to about 0.5 mole.

Park’s empirical formula for the primary particle can include Co in an amount that can range from $0 \leq y \leq 0.5$ mole, exactly as claimed. Ex_1008, ¶28, Ex_1002, ¶214.

9. Claim 9

Park also discloses claim 9, which further recites that cobalt concentration ranges from 0.0 to about 0.3. Park discloses lithium composite oxides having the formula $\text{LiNi}_{0.5}\text{Co}_{0.2}\text{Mn}_{0.2}\text{O}_2$, such that cobalt is 0.2 moles, squarely within the range. Ex_1008, ¶28, Ex_1002, ¶215.

10. Claim 10

Park, alone or in view of Lampe-Onnerud, discloses or renders obvious claim 10, which further recites that the first composition includes 0.8-1 of nickel. Park's empirical formula for the primary particle can include Ni in an amount that can range from $0.5 \leq x \leq 1$ mole, which closely overlaps the claimed range. Ex_1008, ¶28, Ex_1002, ¶216.

All of the examples in Lampe-Onnerud are within the claimed range. For example, this is disclosed in Examples 1 and 2 ($\text{Ni}_{0.8}$), Examples 3, 6-13 ($\text{Ni}_{0.87}$), and 15 ($\text{Ni}_{0.90}$). Ex_1007, 9:60-16:1. It would have been obvious to a POSITA to utilize amounts of nickel at the higher end of Park's range, such as in examples in Lampe-Onnerud, because it was well-understood (and recognized in Park itself) that additional nickel tends to reduce cost. Ex_1002, ¶217.

11. Claim 11

Park, alone or in view of Lampe-Onnerud, discloses or renders obvious claim 11, which further recites that the first and second compositions include about Mn in an amount of about 0.01-0.6 mole.

Park's empirical formula for the core and for the coating can include Mn in an amount that can range from $(0 \leq z \leq 0.5)(2 - (0.9 \leq a \leq 1))$. For example, if $z = 0.5$ and $a = 1$, this computes to 0.5 mole of Mn, within the range. Similarly, in Lampe-Onnerud, Examples 6 and 7 describe synthesis of a base material having 0.05 of manganese in primary particles and the grain boundaries. Ex_1007, 13:1-35.

A POSITA would have understood from Park and Lampe-Onnerud that there will be uniform distribution of manganese in primary particles and grain boundaries, within the claimed ranges of amounts. Ex_1002, ¶220.

12. Claim 12

Park, alone or in view of Lampe-Onnerud, discloses or renders obvious claim 12, which depends from claims 1 or 7 and further recites that the first composition includes nickel in a range of 0.8-1. This limitation is met for at least the same reasons discussed above for dependent claim 10 in this section. Ex_1002, ¶221.

13. Claim 13

Park in view of Lampe-Onnerud renders obvious claim 13, which recites nickel at a concentration of 0.92 moles.

As discussed above, Lampe-Onnerud's empirical formula provides a range of 0.6-1 moles of nickel in the core, and the examples are for 0.8, 0.87, and 0.90 moles of nickel. Ex_1007, Examples. A POSITA would have understood these ranges to reflect average compositions in primary particles and that there will naturally be some variation. It was well-understood that if one were to perform the example in which a

base material is synthesized with 0.90 mole of Ni (Example 15), at least some primary particles, in at least some area in the bulk, will be characterized as having slightly more or less amounts of nickel, and therefore would have an amount of 0.92. Ex_1002, ¶223.

14. Claim 14

Park discloses claim 14, which depends from claims 1 or 7 and recites wide ranges of lithium, nickel, and cobalt in the “first composition.”

Park discloses an empirical formula of $\text{Li}_a(\text{Ni}_x\text{Co}_y\text{Me}_z)_{2-a}\text{O}_2$, where $0.5 \leq x \leq 1$, $0 \leq y \leq 0.5$, $0 < z \leq 0.5$, $0.90 < a \leq 1.15$, which are as follows:

	Claim 14 ranges	Park
Lithium	0.1-1.3	Li_{x1} , where “x1” is 0.9 to 1.15 (Example 1 is 1)
Nickel	0.1-1	Core is 0.425-1.1 (0.5 to 1)(2-(.9 to 1.15))
Cobalt	0-0.5	Core is 0-0.55 (0 to 0.5)(2-(0.9 to 1.15))

Ex_1008, ¶28; Ex_1002, ¶224. This includes $\text{LiNi}_{0.6}\text{Co}_{0.2}\text{Mn}_{0.2}\text{O}_2$, which is squarely within the range for lithium, nickel, and cobalt. *Id.*

15. Claim 15

Park in view of Lampe-Onnerud discloses claim 15, which is dependent from claims 1 or 7 and further recites very wide cobalt concentrations by atomic percentage for the reasons explained in Section VII.C.15. Park discloses the claimed first composition as having Co in an amount that falls within the claimed range. Ex_1008, ¶28, 63, *see also* Section VII.D.1.a, *supra*. Based on the amounts of Co disclosed in

Lampe-Onnerud's coating, a POSITA would have expected the combination of Park and Lampe-Onnerud to result in a Co concentration in the grain boundary that falls within the claimed range. Ex_1003, ¶¶20-21; Ex_1002, ¶178. As discussed above, it would have been obvious to modify Park's cathode material by including the cobalt salt coating of Lampe-Onnerud to enrich Park's grain boundaries by increasing its cobalt concentration to improve the cathode's capacity and safety. See Section VII.D.1.c, *supra*; Ex_1002, ¶225.

16. Claim 16

Park, alone or in view of Lampe-Onnerud, discloses claim 16, which further recites a layer on the surface of the particle. Park discloses that the amorphous lithium salt can be lithium carbonate. Ex_1008, ¶¶25, 31. Moreover, Lampe-Onnerud discloses applying an LiCoO_2 -based coating, which is an oxide, as recited in the claim. The coating also can include Al, B, or Zr, as claimed. Ex_1007, 2:57-67 ("... the coating on the core has ... at least one element selected from the group consisting of ... boron, aluminum ... and zirconium."). Ex_1002, ¶226.

It would have been obvious to include the coating from Lampe-Onnerud with Park's particle for purposes of further isolating the particle from the electrolyte, as Park discloses. Ex_1008, ¶14 (describing that it is advantageous "to reduce the specific surface area of the positive electrode active material that is exposed to the electrolyte"). Ex_1002, ¶227.

17. Claim 17

Park in view of Lampe-Onnerud renders obvious the method of claim 17 for the following reasons:

- a) **“.... combining a lithium compound, a cobalt compound, and a nickel compound to form a mixture;”**

Park discloses adding a lithium compound to a lithium composite oxide such as $\text{Li}_a(\text{Ni}_x\text{Co}_y\text{Me}_z)_{2-a}\text{O}_2$, a nickel compound. Ex_1008, ¶¶12, 28, 63; Ex_1002, ¶229. As described in the introduction to Ground 2 and for claim 1, it would have been obvious to add a cobalt compound along with lithium salt to enrich the grain boundary, in combination with the disclosures of Lampe-Onnerud, to further improve the stability and of the material. *See* Section VII.D *supra*. A POSITA would have understood that the combination of the Park particle with the cobalt gradient of Lampe-Onnerud would improve the “capacity, cyclability and safety” of the cathode active material. Ex_1007, 2:16-28, 9:43-45; Ex_1002, ¶231.

- b) **heating the mixture at about 30 to about 200° C. to form a dried mixture;”**

Park in view of Lampe-Onnerud renders this method step obvious. Lampe-Onnerud discloses that the base material is mixed with a cobalt-based material in distilled water and then heated and dried. Ex_1007, 13:20-26. It would have been obvious to add the cobalt salt for enriching the grain boundary by a wetting process. Ex_1002, ¶191. In Lampe-Onnerud, a POSITA would have understood that the

“evaporated off on a hot plate” for drying the mixture would be in a temperature range of about 30 to about 200° C. Ex_1007, 13:20-26; Ex_1002, ¶232.

- c) “heat treating the dried mixture at about 200 to about 500 °C. for about 0.1 to about 5 hours;”**

Continuing with Example 1, Lampe-Onnerud discloses that after evaporation, the dried mixture is sintered in a heat ramp at 5°C/minute to 450°C and held for 1 hour. Ex_1007, 13:26-30. This is within the claimed temperature range and timing.

- d) “heat treating at 600 to about 900 °C. for about 0.1 to about 10 hours to manufacture the particle”**

Park discloses a final heating step at 800°C or 900°C, which are both within the claimed range. Ex_1008, ¶64-65. Similarly, Example 1 of Lampe-Onnerud discloses a final heating step, this time ramping by 2°C/minute to 750°C and holding for 1 hour, also within the claimed temperature range and timing. Ex_1007, 13:30-31.

- e) “wherein the particle comprises”**

Claim 17(e) then recites the product of the process, which repeats the limitations for the particle in claim 1. The limitations regarding the material are disclosed in both Park and Lampe-Onnerud, at least for the reasons set forth in the discussion at Sections VII.D.1.a-c (regarding claim 1).

E. GROUND 3: TAKAGI ANTICIPATES CLAIMS 1-9, 11, AND 14-17

Japanese Published Application No. JP 2003-59489A to Takagi was published on February 28, 2003, and qualifies as 35 U.S.C. §§ 102(b) prior art. Takagi pertains

to a battery cathode material having a stabilized crystal structure to improve performance.

Takagi and the '317 patent provide similar accounts for why conventional lithium nickel oxide cathode materials are deficient. Takagi explains that these materials experience “cracking at the bonding interface caused by repeated charge/discharge cycles.” Ex_1010, ¶6. Takagi also notes that lithium cobalt oxide materials are expensive. *Id.*, ¶3. Takagi also framed the problem to be solved as finding a material for a rechargeable battery “that is low in cost and has excellent cycle characteristics.” *Id.*, ¶¶7-8.

Just like the '317 patent, Takagi recognized that a solution is to synthesize cathode materials such that the portion “near the surface” of the primary particles, which Takagi also refers to as the “bonding interface,” has higher cobalt concentration than areas inside. *Id.*, ¶¶9-10. A POSITA would have understood that a bonding interface is a grain boundary. Ex_1002, ¶245. Takagi explicitly recognized that this is a way “to effectively use expensive cobalt, leading to cost reduction.” Ex_1010, ¶11.

Because secondary particles are formed of primary particles with increased cobalt at their edges/surfaces, grain boundaries that form at interfaces of adjacent primary particles have increased cobalt concentration. This shores them up to prevent disintegrating and cracking.

Takagi's process causes cobalt to adhere to the primary particle surfaces when they are originally formed, which then become part of the secondary particles' grain boundaries after the final sintering steps. This approach addresses the same problem as in the '317 patent and also results in a greater concentration of cobalt in the grain boundary than in crystallites. Ex_1002, ¶241.

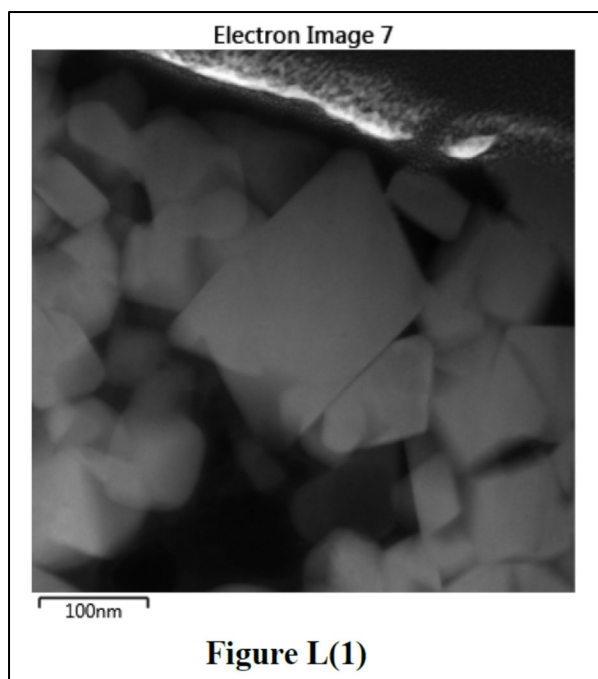
1. Claim 1

- a) “A particle, comprising: a plurality of crystallites comprising a first composition having a layered α -NaFeO₂-type structure and comprising: lithium, nickel, and oxygen”**

Takagi pertains to a cathode material that is “a secondary particle having a plurality of primary particles containing a layered rock salt structure lithium complex oxide containing at least lithium, nickel, and cobalt.” Ex_1010, ¶15.

It was well understood that a “layered rock salt structure” of lithium nickel oxide-based cathode material has a layered α -NaFeO₂-type structure. *See* Ex_1012, 940 (“LiNiO₂, which also forms the α -NaFeO₂ structure”); Ex_1019, 707 (LiCoNiMnO₂ “possess[] the ... α -NaFeO₂-type structure ...”); Ex_1002, ¶243. For example, Zheng also has a lithium nickel cobalt aluminum oxide, specifically, LiNi_{0.8}Co_{0.15}Al_{0.05}O₂, that corresponds to the empirical formula in Takagi, which has “an ordered layered (α -NaFeO₂-type) in the grain interior.” Ex_1011, A357-58. This structure is fundamental to the energy storage capabilities of a battery cathode. Ex_1002, ¶243; Ex_1011, A361.

Petitioner's replication of Takagi Example 5 confirmed that by performing that process there are agglomerated primary particles, or crystallites, similar to those in FIGS. 1 and 10 of the '317 patent. Ex_1003, Figures L(1)-L(2); Ex_1002, ¶244. Image L(1) from Petitioner's replication of Example 5 shows the polycrystalline material with many agglomerated primary particles.

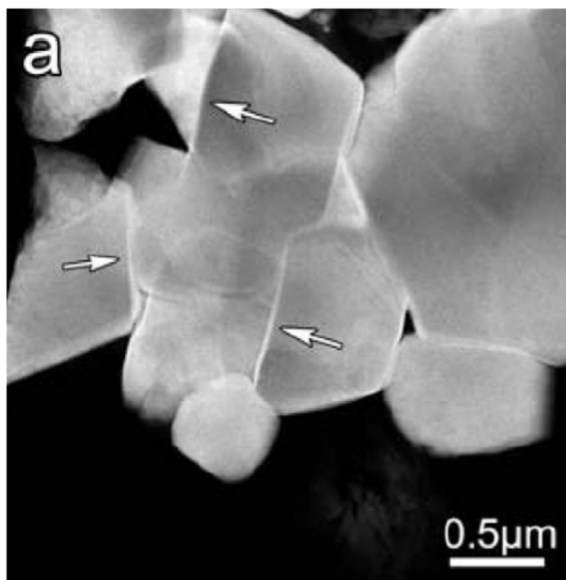


Ex_1003, ¶42, Figure L(1).; Ex_1002, ¶244.

b) “a grain boundary between adjacent crystallites of the plurality of crystallites ...”

As mentioned above, Takagi discloses a material having secondary particles, which have agglomerated primary particles that form grain boundaries at their “bonding interface.” *See, e.g.*, Ex_1010, ¶¶6, 9, 19. As properly construed, a grain boundary is a region located generally at the edges/surfaces of and between adjacent crystallites where they interface with each other. Ex_1002, ¶245.

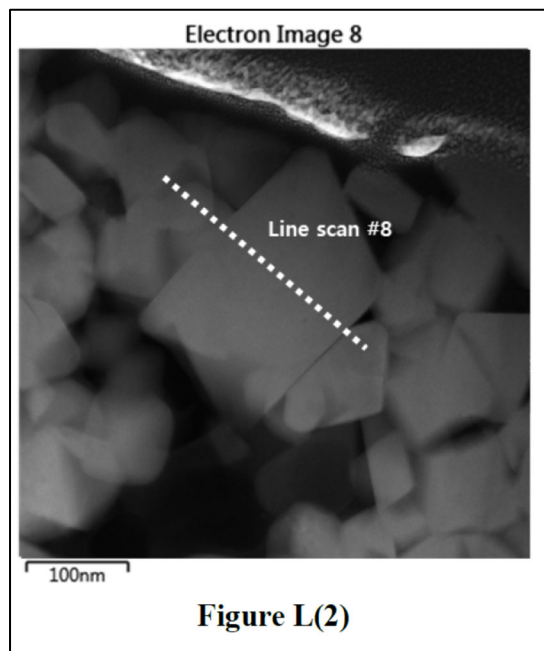
As an example, Zheng illustrates an image of a secondary particle of a lithium nickel cobalt aluminum oxide cathode material, showing the grain boundaries therein.



Ex_1011, FIG 5, A359 (grain boundaries annotated with white arrows in original);
Ex_1002, ¶246.

Petitioner's experimentation of Example 5 of Takagi yielded secondary particles with grain boundaries, analogous to those depicted in '317 patent FIG. 10.
Ex_1002, ¶247.

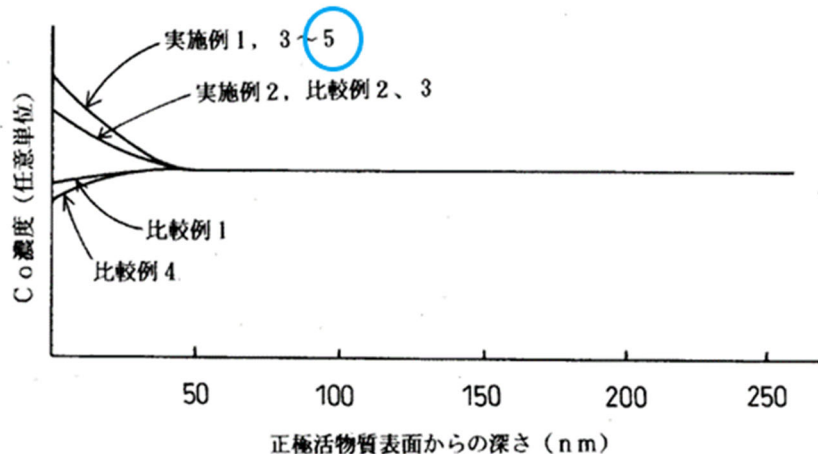
Additionally, the Petitioner's Takagi experiment illustrates grain boundaries at the edges of primary particles:



Ex_1003, Figure L(1) (line scan #8 crosses two grain boundaries); Ex_1002, ¶247.

- c) **“... comprising a second composition having the layered α -NaFeO₂-type structure, a cubic structure, or a combination thereof, wherein a concentration of cobalt in the grain boundary is greater than a concentration of cobalt in the crystallites.”**

Takagi synthesizes primary particles such that the portion “near the surface,” which the reference also refers to as the “bonding interface” or grain boundary, has higher concentration of cobalt relative to the primary particle’s “inside.” Ex_1010, ¶10, 11, 13, 14, 19. This is shown in Takagi FIG. 1, Example 5, where concentration of cobalt is highest at the outer surface of the primary particles (at 0 nm at the left) and decreases toward the inside. Ex_1002, ¶247.

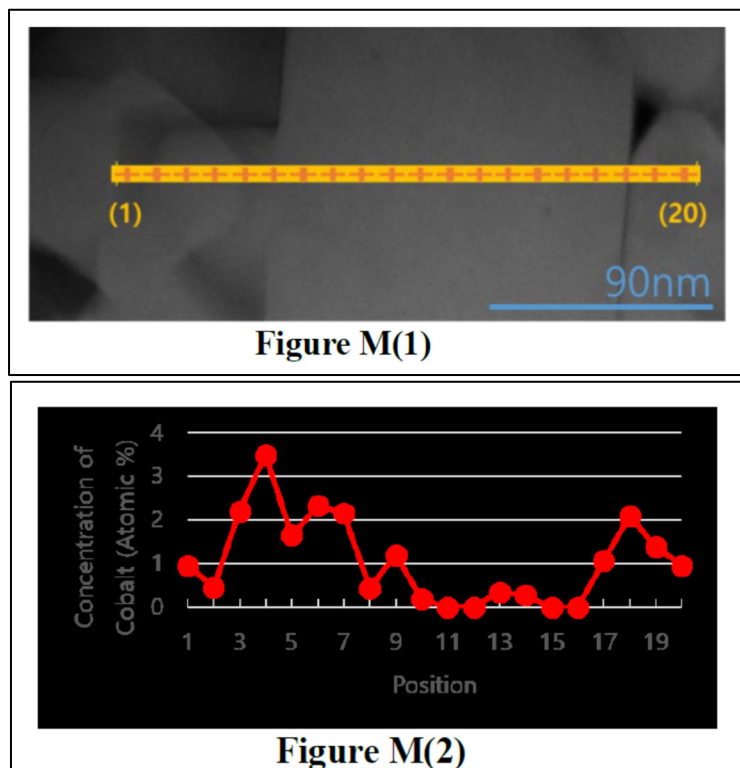


Ex_1010, FIG. 1 (annotated). Takagi explains that “setting the cobalt concentration in the portion near the surface of the primary particles to be high enables suppression of occurrence of cracking at the bonding interface of the primary particles and thus is preferable.” Ex_1010, ¶19.

As such, two compositions exist: a “first composition” at the inner portion of the primary particle and a different “second composition” at the surface portion that forms grain boundaries, which has higher concentration of cobalt. *Id.*

The composition in the surface layer/grain boundary might be the same layered α - NaFeO_2 -type structure as the interior in some regions or comprise a cubic structure in others, which corresponds to the disordered “second composition” of claim 1. Ex_1002, ¶251.

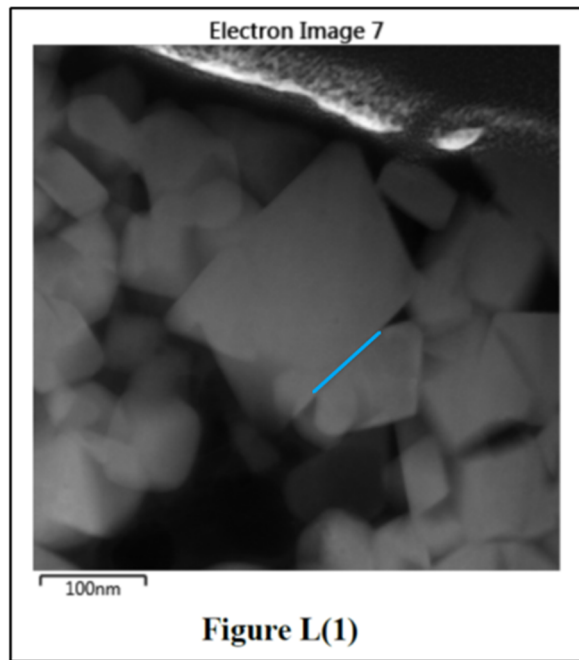
Petitioner’s replication of Takagi Example 5 confirmed that there is higher concentration of cobalt in grain boundaries.



Ex_1003, ¶41-43, FIGS. M(1)-M(2) (for line scan #8 of FIG. L(2), *supra*). The regions where the grain boundary is crossed (positions 4 and 18) have higher cobalt concentration than interiors of adjacent primary particles, or crystallites (positions 2 and 11-16). Ex_1002, ¶253. At grain boundaries, there is comparatively more cobalt and less nickel, and so they are a “second composition.” *Id.*

2. Claim 2

Claim 2 is anticipated by Takagi. In Petitioner’s experiments, the resultant SEM images show primary particles having irregular, non-spherical shapes. Ex_1003, Figure L(1); Ex_1002, ¶247. The edges of at least some approximate straight lines, and therefore they are rectilinear. Ex_1002, ¶254.

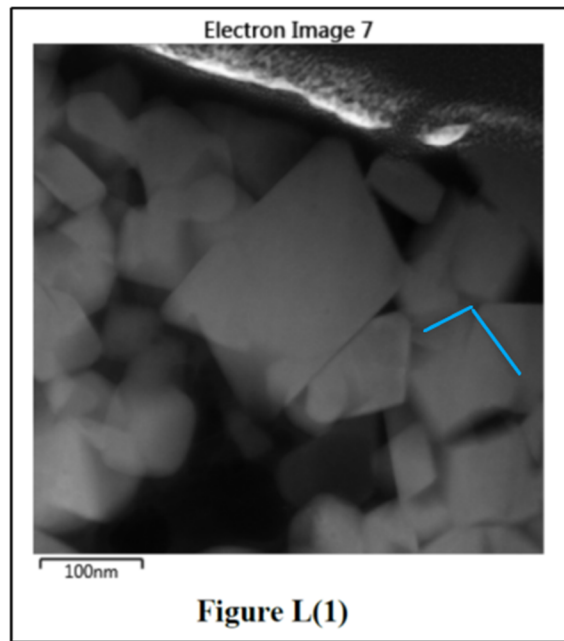


3. Claim 3

Claim 3 is anticipated by Takagi at least for the same reasons set forth for Grounds 1 and 2, Claim 3. The '317 patent relies on FIG. 1 as support, and Petitioner's replication of Takagi Example 5 yielded virtually the same image, indicating that grain boundaries are oriented in various random directions, which are different than a tangent at the outer surface. Ex_1002, ¶255.

4. Claim 4

Claim 4 is anticipated by Takagi at least for the same reasons set forth for Ground 1, Claim 4. As can be seen from the image below from Petitioner's experiment of Example 5 from Takagi, two grain boundaries are highlighted on the same crystallite, intersecting at an angle according to the crystallite's shape. Ex_1002, ¶255.



Ex_1003, ¶42, Figure L(1) (annotated).

5. Claim 5

Claim 5 defines the length of the grain boundary as being “parallel to a surface of an adjacent crystallite” and requires both the length and the average length of the grain boundary to be from “about 50 to about 1000 nanometers.”

Takagi discloses that the primary particles can have an average diameter of “about 500 nm,” which can correspond with the length of a side and be coincident with the grain boundary length. Ex_1010, ¶54. Ex_1002, ¶258. Therefore, Takagi discloses grain boundary lengths within the claimed ranges.

Claim 5 also defines the thickness of the grain boundary as being “perpendicular to the crystallite surface” and requires both the thickness and average thickness of the grain boundary to be from “about 1 to about 200 nanometers”. Takagi

discloses that the “surface” where the cobalt concentration is increased is about 50 nm, which also is within the claimed range. Ex_1010, ¶52; Ex_1002, ¶259.

6. Claim 6

The “second composition” in Takagi falls within the claimed ranges. Takagi discloses that the general formula is $\text{LiNi}_{1-x-y}\text{Co}_x\text{M}_y\text{O}_2$ where X is preferably 0.01 or more and 0.5 or less, and Y is preferably 0.01 or more and 0.2 or less. From this it can be deduced that there is:

	Claim 6 ranges	Takagi Grain Boundary
Lithium	0.1-1.3	1
Nickel	0.1-1	0.2-0.98 (Ni_{1-x-y} where X is preferably 0.01 or more and 0.5 or less, and more preferably 0.3 or less, and Y is preferably 0.01 or more and 0.2 or less) The grain boundary region will have a lower amount of nickel than the average empirical formula amount, which will still be within the range
Cobalt	0.02-0.99	On average, 0.01-0.5 (Co_x , where X is preferably 0.01 or more and 0.5 or less) The grain boundary region will have a greater amount of cobalt than the average empirical formula amount, so the amount is within the range
Oxygen	1.7-2.3	2

Ex_1002, ¶260.

7. Claim 7

Takagi anticipates claim 7, as the reference discloses that the cathode material preferably includes a range of 0.01 to 0.2 of Fe, Mn, or Al. Ex_1010, ¶18. That

amount will be evenly distributed in the bulk and at the surface, and so the range corresponds to both the first and second compositions as recited in claim 7. Ex_1002, ¶261.

8. Claims 8 and 9

Takagi discloses that the empirical formula includes cobalt that is “preferably 0.01 or more and 0.5 or less,” which within the range recited in claim 8. Ex_1010, ¶18. Ex_1002, ¶262.

Takagi also discloses that the range is “more preferably 0.3 or less,” which is the range recited in claim 9. Ex_1010, ¶18; Ex_1002, ¶263.

9. Claim 11

Takagi discloses the empirical formula can include Mn in a range “preferably 0.01 to 0.2.” That amount will be evenly distributed in the bulk and at the surface, and so the range corresponds to both the first and second compositions as recited in claim 11. Ex_1002, ¶264.

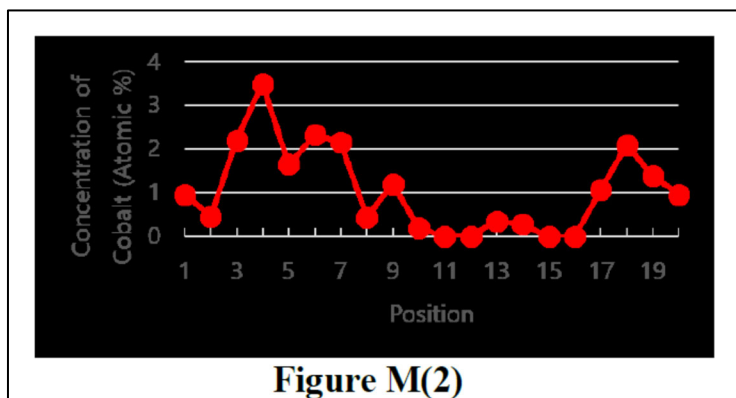
10. Claim 14

Takagi discloses the ranges recited in claim 14 for at least the reasons set forth for claim 6, *supra*.

11. Claim 15

From performing Takagi’s Example 5, as can be seen from FIG. M(2), there are cobalt concentrations in the crystallites that are greater than 0.25% and less than/equal to about 1% (e.g., at positions 1-2, 10, 13-14, and 20), and cobalt concentrations in

the grain boundary between 2% to 3% (at positions 4 and 18), all within the claimed range.



Ex_1003, ¶41-43, FIGS. M(1)-M(2).

12. Claim 16

Takagi teaches that the cathode active material can include a binder having fluorides such as polyvinylidene fluoride. Ex_1010, ¶¶32, 57. As such, Takagi anticipates Claim 16. Ex_1002, ¶266.

13. Claim 17

Takagi discloses the method of claim 17.

First, Takagi combines “raw materials” including a mixture of oxides and hydroxides of “at least lithium, nickel and cobalt” which are the three claimed compounds. Ex_1010, ¶26; Ex_1002, ¶268.

Takagi also discloses three heating steps in the claimed ranges. In example 5 of Takagi, the materials are heated at 200° C for 3 hours, then 500° C for 5 hours, then 700° C for 5 hours. Ex_1010, Table 1, Example 5; Ex_1002, ¶269. These temperatures and times fall exactly within the ranges described in claim 17.

The resulting material has the claimed structure and composition, as set forth in this ground for claim 1, *supra*.

F. GROUND 4: CLAIMS 10, AND 12-13 WERE OBVIOUS OVER TAKAGI

Claims 10 and 12 recite that the “first composition” has 0.8-1 moles of nickel, and claim 13 recites having 0.92 moles of nickel.

Takagi discloses that the general formula is $\text{LiNi}_{1-X-Y}\text{Co}_X\text{M}_Y\text{O}_2$ ($0 < X < 1$; $Y \geq 0$; M is selected from Fe, Mn, and Al), where X is “preferably” 0.01 or more and 0.5 or less, and “more preferably” 0.3 or less,” and that “Y is preferably 0.01 or more and 0.2 or less.” Ex_1010, ¶18. Accordingly, the preferred average for nickel is 0.5-0.98, which overlaps the range for claims 10 and 12.

The claimed ranges are rendered obvious because they overlap with or lie inside those disclosed by Takagi. Takagi’s disclosure of nickel concentration also renders claims 10, 12-13 obvious because it discloses the general conditions based on which a POSITA would have discovered optimum or workable ranges through mere routine experimentations. Moreover, it would have been obvious to optimize concentration of nickel because it was well known to be a result effective variable in that it directly influences the overall cost of the secondary particles as well as cathode capacity. See Ex_1002, ¶271; Ex_1008; Ex_1005. It was also well known that cathodes have 0.8-1 mole of nickel, including around 0.9 mole of nickel in cathode materials. See e.g.,

Ex_1011, A357 ($\text{LiNi}_{0.8}\text{Co}_{0.15}\text{Al}_{0.05}\text{O}_2$); Ex_1022, ¶100 ($\text{Li}(\text{Ni}_{0.85}\text{Co}_{0.15})_{0.93}\text{Al}_{0.07}\text{O}_2$); Ex_1007, 14:1-3 ($\text{LiMg}_{0.01}\text{Ni}_{0.87}\text{Co}_{0.08}\text{Mn}_{0.05}\text{O}_2$).

Nothing in the '317 patent or the intrinsic record establishes that the ranges of claims 10, 12-13 are critical to the operability of the claimed particle.

VIII. THE BOARD SHOULD NOT DENY INSTITUTION UNDER § 325(D)

None of the grounds set forth above were considered by the Examiner during prosecution. Neither Park nor Takagi were identified during prosecution. Lampe-Onnerud was disclosed only after the Office issued a Notice of Allowance, but the reference was not applied.

The application for the '317 Patent was allowed on March 10, 2016. On April 25, 2016, an Information Disclosure Statement (IDS) citing WO 2002/103824, which is the WIPO counterpart to Lampe-Onnerud. Ex_1025, 17. However, even though Lampe-Onnerud is the same assignee as the '317 patent, the applicant did not disclose this or acknowledge that Lampe-Onnerud discloses a process that is virtually the same as that in the '317 patent specification. The Examiner also did not have the benefit of the Petitioner's replication of the process in Lampe-Onnerud, which confirms the presence of increased cobalt concentration in the grain boundaries and provides STEM and STEM-EDS images that match those in the '317 patent. Accordingly, the Office erred in a manner material to the patentability of the challenged claims by not applying the teachings of Lampe-Onnerud, and the citation

of Lampe-Onnerud in an IDS should not serve as a basis for denial of institution here. *Advanced Bionics, LLC v. Med-El Elektromedizinische Geräte GmbH*, IPR2019-01469, Paper 6 at 8 (precedential) (Feb. 13, 2020); *Becton, Dickinson and Co. v. B. Braun Melsungen AG*, IPR2017-01586, Paper 8 at 16-18, 24-25 (PTAB Dec. 15, 2017). These reasons warrant consideration of Lampe-Onnerud in the above-asserted grounds during trial here. *Advanced Bionics*, at 8-9.

IX. CONCLUSION

For the foregoing reasons, Petitioner respectfully requests *inter partes* review of claims 1-17 of the '317 Patent.

X. MANDATORY NOTICES UNDER 37 C.F.R. § 42.8(A)(1)

Pursuant to 37 C.F.R. § 42.8(a)(1), the mandatory notices identified in 37 C.F.R. § 42.8(b) are provided below as part of this petition.

A. 37 C.F.R. § 42.8(b)(1): Real Parties-In-Interest

The real party-in-interest is POSCO Future M Co., Ltd.

B. 37 C.F.R. § 42.8(b)(2): Related Matters

Pursuant to 37 C.F.R. § 42.8(b)(2), Petitioner is not aware of any infringement lawsuit involving the '317 Patent.

C. 37 C.F.R. § 42.8(b)(3), (4): Counsel And Service Information

Petitioner provides the following designation of lead and back-up counsel:

Lead Counsel	Back-up Counsel
Michael A. Oblon (Reg. No. 42,956) JONES DAY 51 Louisiana Ave., N.W. Washington, D.C. 20001-2113 Telephone: (202) 879-3815 Facsimile: (202) 626-1700 moblon@jonesday.com	Matthew Johnson (Reg. No. 59,108) JONES DAY 500 Grant Street, Suite 4500 Pittsburgh, Pennsylvania 15219-2514 Telephone: (412) 391-3939 Facsimile: (412) 394-7959 mwjohnson@jonesday.com
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Pursuant to 37 C.F.R. § 42.10(b), a Power of Attorney for Petitioner accompanies this petition. Please address all correspondence to lead and back-up counsel at the addresses above. Petitioner also consents to electronic service by email at the email addresses listed above.

Dated: July 29, 2024

Respectfully Submitted,

/s/ Michael A. Oblon

Michael A. Oblon (Reg. No. 42,956)
Counsel for Petitioner

CERTIFICATE OF SERVICE

The undersigned hereby certifies that a copy of the foregoing petition for *Inter Partes* Review of U.S. Patent No. 9,391,317, including all Exhibits, is being served on July 30, 2024, via Express Mail delivery directed to the attorney of record for the patent at the following address:

DINSMORE & SHOHL LLP
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SUITE 1900
TROY, MI 48084

Dated: July 29, 2024

Respectfully Submitted,

/s/ Michael A. Oblon

Michael A. Oblon (Reg. No. 42,956)
Counsel for Petitioner

CERTIFICATE OF WORD COUNT UNDER 37 C.F.R. § 42.24(A)

I, the undersigned, do hereby certify that the attached petition contains 13,971 words, as measured by the Word Count function of Microsoft Word. This is less than the limit of 14,000 words as specified by 37 C.F.R. § 42.24(a)(1)(i).

Dated: July 29, 2024

Respectfully Submitted,

/s/ Michael A. Oblon

Michael A. Oblon (Reg. No. 42,956)
Counsel for Petitioner

APPENDIX OF EXHIBITS

EXHIBIT	TITLE
1001	U.S. Patent No. 9,391,317 (“the ’317 Patent”)
1002	Expert Declaration of Doron Aurbach, Ph.D
1003	Declaration of Hyunkuk Noh, Ph.D
1004	Prosecution file history for U.S. Patent No. 9,391,317
1005	Prosecution file history for U.S. Patent No. 9,209,455
1006	U.S. Provisional Application No. 61/575,115
1007	U.S. Pat No. 6,921,609 to Lampe-Onnerud et al., issued July 26, 2005 (“Lampe-Onnerud”)
1008	U.S. Publ. App. No. 2011/0143209 to Park et al., published June 16, 2011, filed December 1, 2010, and issued as U.S. Patent No. 8,586,247, claiming priority to Prov App. No. 61/285,785 filed December 11, 2009 (“Park”)
1009	Japanese Published Application No. JP 2003-059489 to Takagi et al, published February 28, 2003 (“Takagi”)
1010	Certified translation of JP 2003-059489 (“Takagi”)
1011	Zheng et al., Microstructural Changes in $\text{LiNi}_{0.8}\text{Co}_{0.15}\text{Al}_{0.05}\text{O}_2$ Positive Electrode Material During the First Cycle, Journal of the Electrochemical Society, 158 (4) A357-A362 (2011) (Published February 9, 2011) (“Zheng”)

1012	Fergus, Recent Developments in Cathode Materials For Lithium Ion Batteries, <i>Journal of Power Sources</i> 195 (2010) 939-954 (“Fergus”)
1013	Kalyani et al., Various Aspects of LiNiO ₂ Chemistry: A Review, <i>Science and Technology of Advanced Materials</i> 6 (2005) 689-703 (“Kalyani”)
1014	Volovich et al., Percolation Models of Grain Boundary Wetting in Polycrystalline Materials, <i>Russian Journal of General Chemistry</i> , 2008, Vol. 78, No. 11, pp. 2182-2190 (“Volovich”)
1015	Wittingham, Lithium Batteries and Cathode Materials, <i>American Chemical Society, Chemical Reviews</i> 2004, Vol. 104, No. 10, pp. 4271-4301 (“Wittingham”)
1016	Ghiji et al., A Review of Lithium-Ion Battery Fire Suppression, <i>Energies</i> 2020, 13, 5117 (“Ghiji”) http://doi.org/10.3390/en13195117
1017	Muto et al., Capacity-Fading Mechanisms of LiNiO ₂ -Based Lithium Ion Batteries, <i>Journal of the Electrochemical Society</i> , 156 (5) A371-A377 (2009) (“Muto”)
1018	Liu et al., Comparison of Structure and Electrochemistry of Al- and Fe-doped LiNi _{1/3} Co _{1/3} Mn _{1/3} O ₂ , <i>Electrochimica Acta</i> 51 (2006) 4199-4203 (“Liu”)
1019	Ellis et al., Positive Electrode Materials for Li-Ion and Li-Batteries, <i>Chemistry of Materials Review</i> , 2010, 22, 691-714 (“Ellis”)
1020	Turner et al., Surface Analysis: X-ray Photoelectron Spectroscopy, Auger Electron Spectroscopy, and Secondary Ion Mass Spectrometry, <i>Anal Chem</i> , 1982, Vol. 54, pp. 293-322 (“Turner”)

1021	U.S. Published Patent Appl. No. 2009/0104530 to Shizuka et al
1022	U.S. Publ. App. No. 2011/0033750 to Hosokawa et al., published February 10, 2011, filed March 3, 2010 (“Hosokawa”)
1023	Japanese Published Application No. JP 2010/047466 A (issued as Japanese Patent JP 5451228 B2), Lithium Transition Metal Oxide with Layer Structure, published March 4, 2010 (“Kagei”)
1024	Certified translation of Kagei excerpt
1025	File History for Lampe-Onnerud
1026	US Provisional Application 61/285,785
1027	U.S. Pat No. 7,381,496 to Onnerud et al., issued June 3, 2008 (“Onnerud”)
1028	Declaration of Michael A. Oblon