

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

POSCO FUTURE M CO., LTD.,
Petitioner,

v.

TIAX LLC,
Patent Owner.

Case No. – Not Yet Assigned
Patent 9,391,317

DECLARATION OF HYUNKUK NOH, PhD

1. My name is Hyunkuk Noh, PhD. I am over the age of twenty-one (21) years, of sound mind and capable of making the statements set forth in this Declaration. I am competent to testify to matters set forth herein. All the facts and statements contained herein are within my personal knowledge and they are, to the best of my knowledge, true and correct.

2. I have been employed by POSCO Future M. Co. Ltd. (“POSCO”) since 2019. My current title is Senior Principal Researcher, which I have held for the last two years. My employment and compensation are in no way dependent upon the outcome of this proceeding.

3. My responsibilities and duties as a laboratory researcher include testing and analyzing samples of cathode active materials of lithium ion batteries, including competitor products. Such analysis often includes measuring the concentration of specific elements in specific locations of primary particles of cathode active materials. I have training and experience in the measurement and analysis of elemental component concentration. I have performed research and development on cathode active materials for over 4 years at POSCO and over 10 years in my previous job. I have experience using and training others to use analytical and testing equipment, such as with focused ion beams, scanning transmission electron microscopes, and energy dispersive (X-ray) spectroscopy built into scanning transmission electron microscopy, throughout my studies and professional career.

4. I earned my doctorate degree in Chemical Engineering specializing in catalysis and environmental engineering from Pohang University of Science and Technology in 2005.

5. I was asked to reproduce and analyze Example 1 of U.S. Patent No. 6,921,609 (the “Lampe-Onnerud patent”), Examples 1 and 2 of U.S. Patent No. 9,391,317 (the “’317 patent), Example 5 of Japanese Published Application 2003-59489A (“Takagi”), and Examples 1 and 7 of U.S. Patent No. 7,381,496 (the “Onnerud patent”).

6. I performed these experiments with lab assistants working under my direct supervision

at the POSCO Gumi Cathode Material Plant in Korea. To perform characterization, I provided material to Gumi Electronics and Information Technology Research Institute (“GERI”) in Korea. I was present at the GERI lab to monitor the work while characterization was being performed. The characterization was performed using HELIOS 5 UX focused ion beam (“FIB”) by Thermo Fisher, JEM-ARM200F scanning transmission electron microscope (“STEM”) by JEOL companies, and X-Max energy dispersive X-ray spectroscopy (“EDS”) built into a scanning transmission electron microscope by Oxford.

I. Replication of Example 1 of the Lampe-Onnerud patent

7. According to the Lampe-Onnerud patent, the purpose of the experiment in Example 1 is to synthesize a 10% (mol/mol) LiCoO_2 gradient active substance on $\text{LiNi}_{0.8}\text{Co}_{0.2}\text{O}_2$.

8. The $\text{LiNi}_{0.8}\text{Co}_{0.2}\text{O}_2$ core was manufactured by POSCO Future M. LiNO_3 was purchased from Samchun Chemical, and $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was purchased from Junsei Chemical.

9. The manufacture of the specimen was performed between April 22-25, 2024.

10. **Testing Method:** The left column includes the steps disclosed in Example 1 of the Lampe-Onnerud patent. The right column of the table below includes the testing method carried out under my supervision. The weight of the precursors used in our testing method corresponds to 30% of the weight of the precursors used in Example 1 of the Lampe-Onnerud patent.

Example 1 of Lampe-Onnerud patent	Steps Taken Under My Supervision
The following were dissolved in 150 ml distilled H_2O : ○ 7.06 g LiNO_3 and ○ 29.69 g $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	Step 1: The following precursors were dissolved in 45 ml of distilled water ○ 2.12 g of LiNO_3 and ○ 8.91 g of $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$

100 g N-82 ($\text{LiNi}_{0.6}\text{Co}_{0.2}\text{O}_2$) to the mixture	Step 2: ¹ 30 g of $\text{LiNi}_{0.8}\text{Co}_{0.2}\text{O}_2$ were mixed into the mixture resulting from Step 1.
The H ₂ O was evaporated off on a hot plate during stirring.	Step 3: The mixture resulting from Step 2 was then stirred on a hot plate until the water evaporated.
The obtained dried cake was ground for 5 minutes in the mortar grinder.	Step 4: The dry substance obtained from Step 3 was subjected to grinding for 5 minutes.
The dried, grinded cake was then placed in an alumina crucible, and sintered under the following heating profile: ○ ramp 5° C/minute to 450° C, hold at 450° C for 1 hour, ○ ramp 2° C/minute to 750° C, and hold for 1 hour ○ The sample was then allowed to cool naturally to room temperature.	Step 5: The grinded material of Step 4 was then placed into alumina crucible and sintered as follows: ○ Heating at 5 °C/min to 450 °C, then maintaining the temperature at 450 °C for 1 hour. ○ Then heating at 2 °C/min from 450 °C to 750 °C, then maintaining the temperature at 750 °C for 1 hour. ○ Then cooling to room temperature.
The sample was ground for 5 minutes to break up agglomerates	Step 6: The material prepared in Step 5 was then grinded for 5 minutes (“final product”).

11. **Test Results:** After the final product was collected, it was analyzed using FIB, STEM and STEM-EDS at the GERI laboratory on April 24, 2024.

12. Using the FIB, thin samples of about 80-100 nm thickness were produced by ion milling the final sample produced by the method listed above. The shape and structure of the thin samples were then analyzed using the STEM under 200 kV of acceleration voltage. The compositions of the thin samples were then analyzed using EDS built into the STEM.

13. I attended the GERI laboratory when analyzing STEM and STEM-EDS and identified line scan locations for analysis of cobalt concentrations.

14. **Figure A(1)** (on the left) is a true and accurate STEM image of a section of the final product prepared under my supervision. In **Figure A(2)**, on the right, I added cross-hairs for easier analysis of the scale and traced blue lines to more clearly show the primary particles from Figure A(1).

¹ While the Lampe-Onnerud patent discloses that the atomic ratio of Ni is 0.6, we believe that this is a typographical error and, as such, used 0.8 Ni in our experiment.

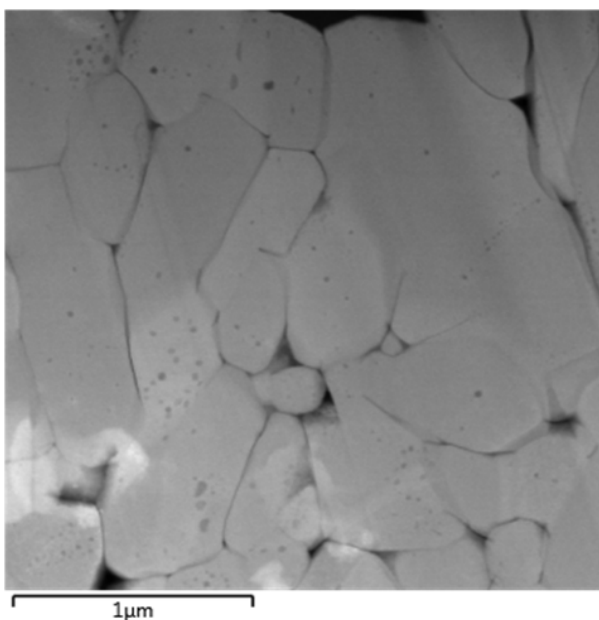


Figure A(1)

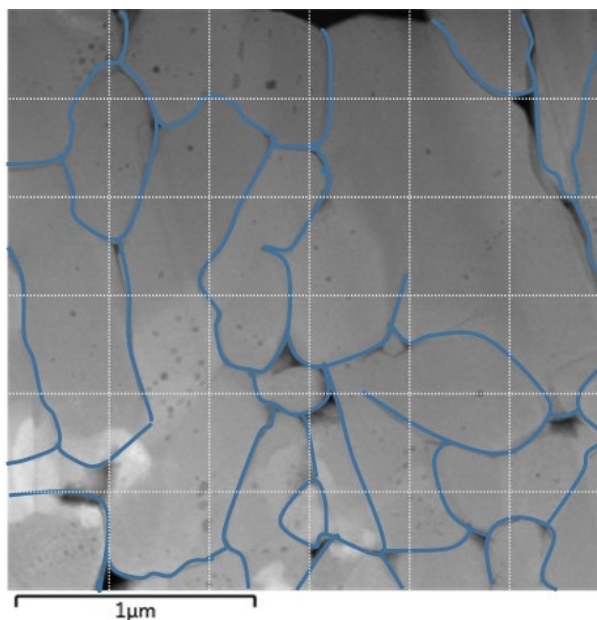


Figure A(2)

15. **Figure B(1)** below is a true and accurate STEM-EDS elemental analysis of the same excerpt of the final product illustrated in Figure A(1). The red dots in the analysis indicate the detected presence of cobalt. **Figure B(2)** is the same image with the blue lines from Figure A(2) shown as white dotted lines.

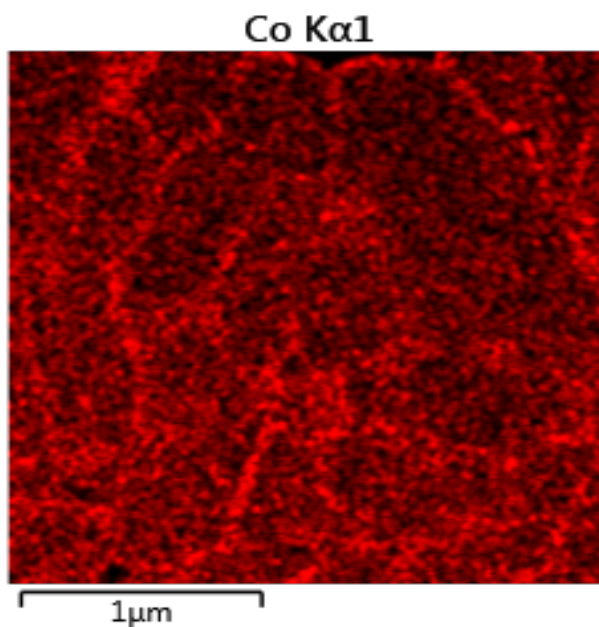


Figure B(1)

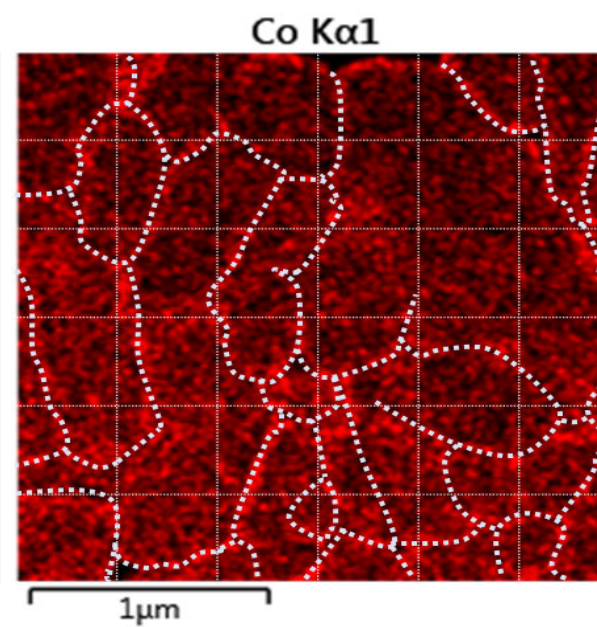


Figure B(2)

16. **Figure C(1)** is the image of Figure A(1) marked with “Line scan 2,” indicating a set

of locations that I selected on the material across which the EDS elemental analysis line scan was performed to measure the concentration of cobalt.

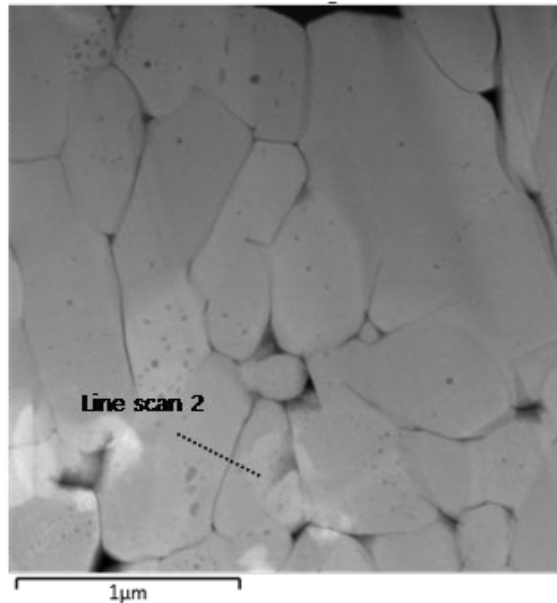


Figure C(1)

17. **Figure C(2)** shows 20 locations analyzed for measurement of the concentration of cobalt in Line scan 2 shown in **Figure C(1)**, each marked with an “S” on the yellow line.



Figure C(2)

18. The gap between each of the 20 locations in **Figure C(2)** was set at about 20 nm. Based on Fig C(2), we calculated the thickness of the Co-enriched grain boundary to be from 40-60 nm.

19. **Figure C(3)** shows the lengths of several grain boundaries from the image in Figure A(1), measured using a computer program called ImageJ. From these measurements, the average length of the grain boundaries is approximately 333.5 nm.

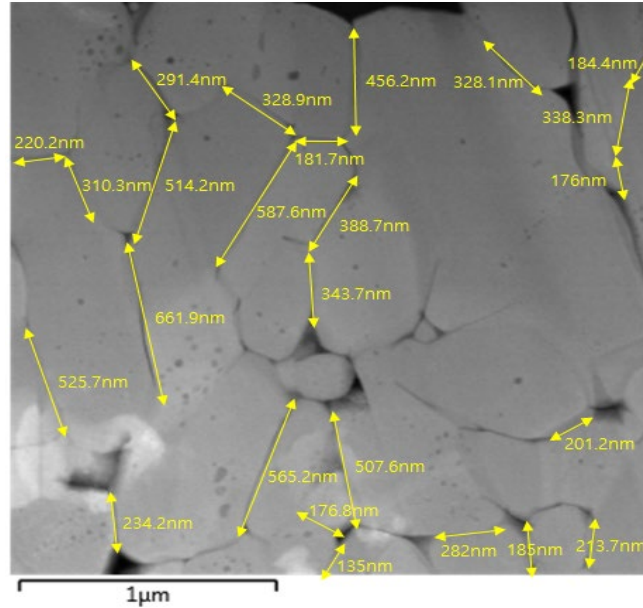


Figure C(3)

20. **Figure D(1)** represents a graph showing the concentration of Cobalt (as atomic %) from the STEM-EDS analysis performed at the locations shown in **Figure C(2)**.

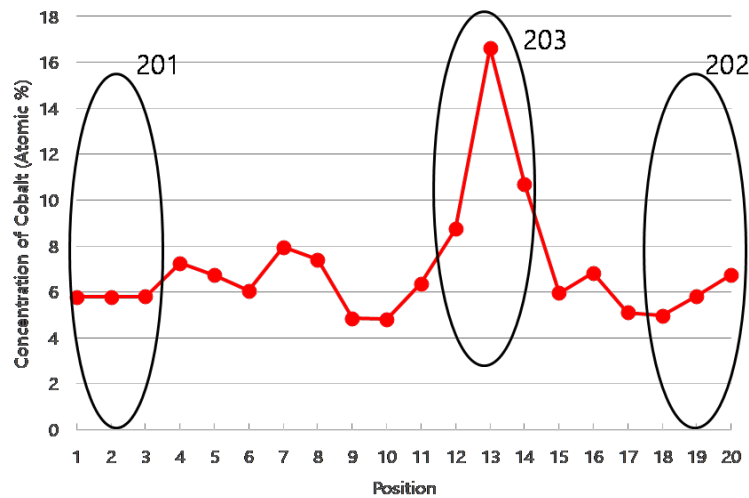


Figure D(1)

21. As seen in **Figure D(1)**, the experimental results confirmed that the concentration of Co in the areas marked as 201 and 202 in the bulk of the crystals was in the range of about 4 to about 8 atomic %, and the Co concentration in the grain boundary between the crystals (spot marked as 203) was in the range of about 8 to about 18 atomic %.

22. The crystal structure of the bulk and the grain boundaries were identified using FFT (Fast Fourier Transform) of the High Resolution-STEM (HR-STEM) in **Figures E, F, and G**.

23. **Figure E** is an image showing regions analyzed by the FFT of HR-STEM of the sample prepared under my supervision. The red square represents the region analyzed for the crystal structure at the grain boundary while the yellow square represents the region analyzed for the crystal structure within the bulk.

24. **Figure F** is an FFT pattern of the red square region identified in **Figure E** as representing the grain boundary, showing a cubic structure at the grain boundary.

25. **Figure G** is an FFT pattern of the yellow square region identified in **Figure E** as representing the bulk. The (003) plane in the FFT pattern indicates an α -NaFeO₂ type structure.

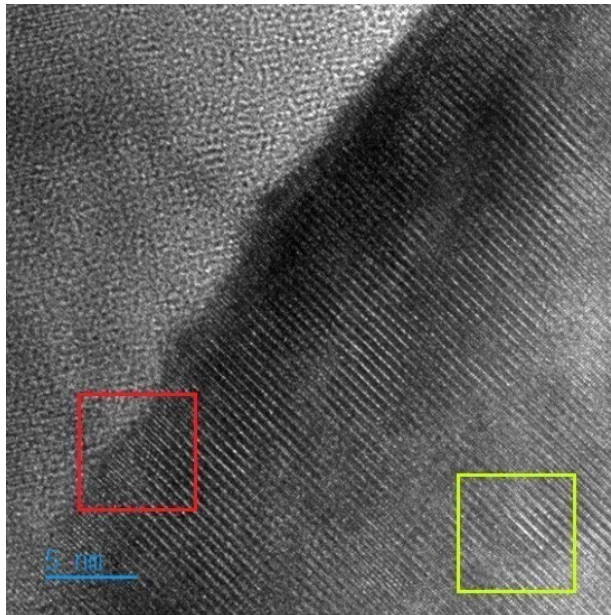


Figure E

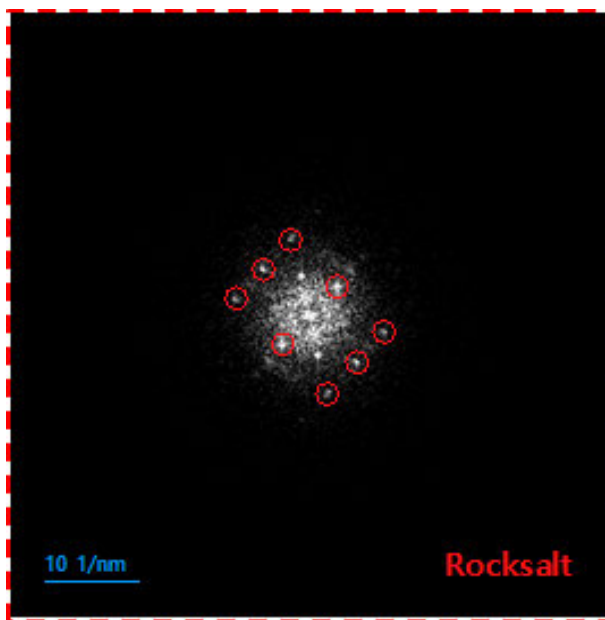


Figure F

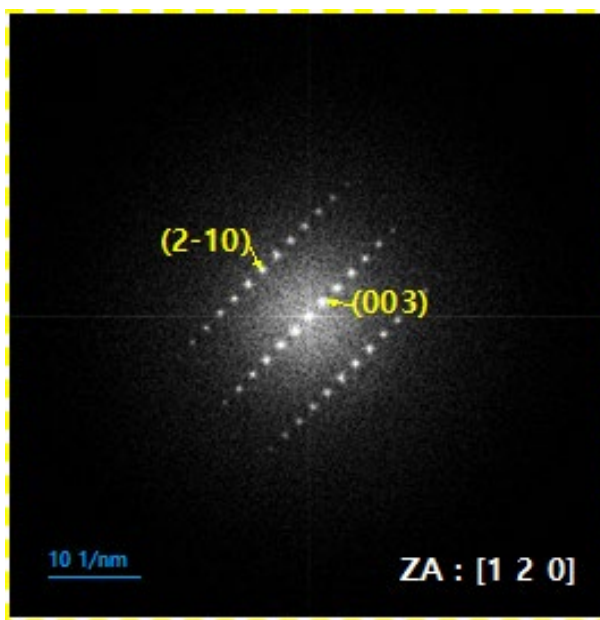


Figure G

II. Replication of Examples 1 and 2 of the '317 patent

26. The purpose of this experiment is to manufacture $\text{Li}_{1.05}\text{Mg}_{0.025}\text{Ni}_{0.92}\text{Co}_{0.08}\text{O}_{2.05}$ of Example 1 and enrich its grain boundaries with cobalt using the steps of Example 2 to produce $\text{Li}_{1.01}\text{Mg}_{0.024}\text{Ni}_{0.88}\text{Co}_{0.12}\text{O}_{2.03}$, and then to analyze the shape and composition of the resulting particles.

27. For this experiment, $\text{Ni}_{0.92}\text{Co}_{0.08}(\text{OH})_2$ was manufactured by POSCO Future M. LiOH was manufactured by Kangwon E-solution, $\text{Mg}(\text{OH})_2$ and LiNO_3 were purchased from Samchun Chemical, and $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was purchased from Junsei Chemical.

28. **Testing Method:** The left column includes the steps disclosed in Examples 1 and 2 of the '317 patent. The right column of the table below includes the testing method carried out under my supervision. The weight of the precursors used in our testing method represents about 50% of the weight of the precursors used in Example 1 of the '317 patent and 10% of the weight of the precursors used in Example 2 of the '317 patent, with all component concentrations adjusted to be stoichiometrically consistent with the disclosure in the '317 patent.

Example 1 of the '317 patent	Steps Taken Under My Supervision
$\text{Li}_{1.05}\text{Mg}_{0.025}\text{Ni}_{0.92}\text{Co}_{0.08}\text{O}_{2.05}$ was prepared by mixing the following: ○ 242.9 g $\text{Li}(\text{OH})_2$ ○ 14.8 g $\text{Mg}(\text{OH})_2$ ○ 35.0 g LiNO_3	Step 1:² The following precursors were mixed by shaking in a 1 L bottle. ○ 129.16 g of LiOH ○ 7.86 g of $\text{Mg}(\text{OH})_2$, ○ 18.59 g of LiNO_3
In a 1 liter jar, 940.3 g $\text{Ni}_{0.92}\text{Co}_{0.08}(\text{OH})_2$ was added to the mixture. The compounds were mixed by shaking the jar.	Step 2: The resulting mixture was then placed in 1 L bottle containing 499.96 g $\text{Ni}_{0.92}\text{Co}_{0.08}(\text{OH})_2$. Then, the mixture was further mixed by shaking.
The mixed compounds were placed in an alumina crucible and sintered. Sintering was performed by: ○ Heating at a rate of about 5° C per minute to about 450° C, and held at about 450° C for about two hours. ○ The temperature was then raised at about 2° C per minute to about 700° C, and held for about six hours. ○ The sample was then allowed to cool naturally to room temperature.	Step 3: After shaking the mixture, it was sintered in alumina crucible as follows: ○ Heating at 5 °C/min to 450 °C, then maintaining the temperature at 450 °C for 2 hours. ○ Then heating at 2 °C/min from 450 °C to 700 °C, then maintaining the temperature at 700 °C for 6 hours. ○ Then cooling to room temperature.
The cooled sample was ground for about five minutes to break up any agglomerates to provide $\text{Li}_{1.05}\text{Mg}_{0.025}\text{Ni}_{0.92}\text{Co}_{0.08}\text{O}_{2.05}$.	Step 4: The resulting material was then crushed for about 5 minutes (“the crushed material”).
Example 2 of the '317 patent	
0.3 grams of LiNO_3 and 117.4 g of $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ were dissolved in 1000 g of H_2O and heated to 60° C.	Step 5: 0.03 g of LiNO_3 and 11.74 g of $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ were dissolved in 100 g of H_2O and heated to 60 °C.
1000 g of the $\text{Li}_{1.05}\text{Mg}_{0.025}\text{Ni}_{0.92}\text{Co}_{0.08}\text{O}_{2.05}$ of Example 1 was added thereto and the resulting slurry was stirred for 30 minutes.	Step 6: 100 g of the crushed resulting material was added to the solution and stirred for 30 minutes to prepare a slurry.
The slurry was then spray-dried to yield a powder.	Step 7: The slurry was then spray dried to obtain powder.
The resulting powder was placed in an alumina crucible and heated at a rate of: ○ about 5° C per minute to about 450° C, and held at about 450° C for about one hour.	Step 8: The powder was then sintered in alumina crucible as follows: ○ Heating to 450 °C at 5 °C/min, then maintaining at 450 °C for 1 hour.

² While the '317 patent discloses using $\text{Li}(\text{OH})_2$, we believe that this is a typographical error and, as such, we used LiOH in our experiment.

○ The temperature was then raised at about 2° C. per minute to about 700° C, and held for about two hours. ○ The sample was then allowed to cool naturally to room temperature to provide a material having the overall composition $\text{Li}_{1.01}\text{Mg}_{0.024}\text{Ni}_{0.88}\text{Co}_{0.12}\text{O}_{2.03}$.	○ Then heating at 2 °C/min from 450 °C to 700 °C, then maintaining at 700 °C for 2 hours. ○ Then cooling to room temperature.
	Step 9: The final sample was produced and collected (“final product”).

29. **Test Results:** After the final product was collected, it was analyzed using FIB, STEM and STEM-EDS.

30. Using the FIB, thin samples of about 80-100 nm thickness were produced by ion milling the final sample produced by the method listed above. The shape and structure of the thin samples were then analyzed using the STEM under 200 kV of acceleration voltage. The composition of the thin samples was then analyzed using STEM-EDS (EDS attached to the STEM).

31. **Figure H** represents an STEM image of the final product prepared under my supervision, as detailed above, for the replication of Examples 1 and 2 of the '317 patent.

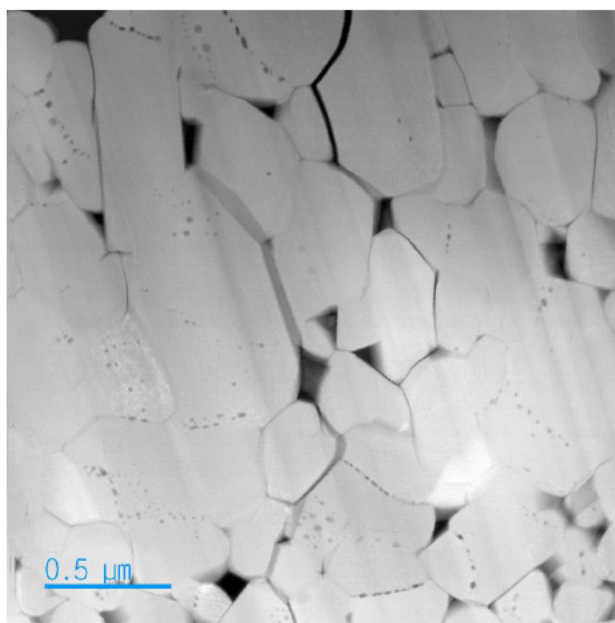


Figure H

32. For comparison, below is Figure 4 of the '317 patent, described as a higher resolution

STEM image of Example 2 particles.

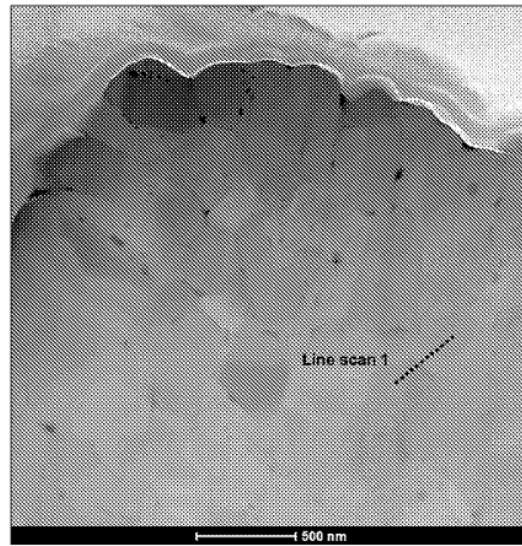


FIG. 4

33. **Figure I** represents an STEM-EDS elemental analysis of the final product. It shows the component analysis of the image in **Figure H**. The areas shaded in red in **Figure I** represent areas where cobalt is concentrated.

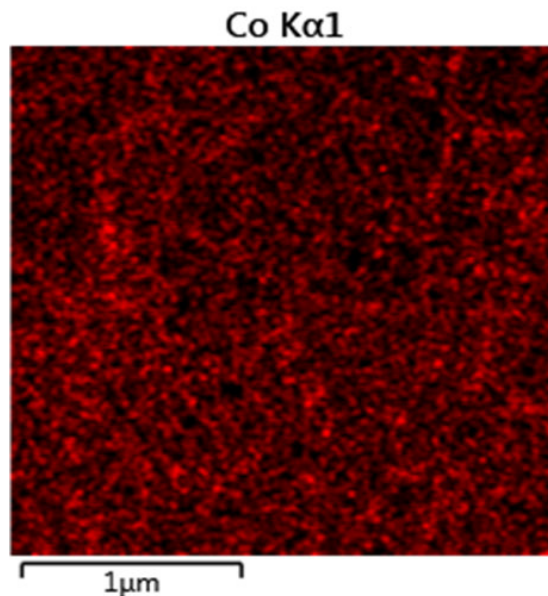


Figure I

34. **Figure J(1)** is **Figure H** additionally marked with Line scan 1, showing a line scan I selected to measure the concentration of cobalt. **Figure J(2)** shows 25 locations analyzed for

measuring the concentration of Cobalt in Line scan 1 in **Figure J(1)**, each marked as “S” in the yellow line.

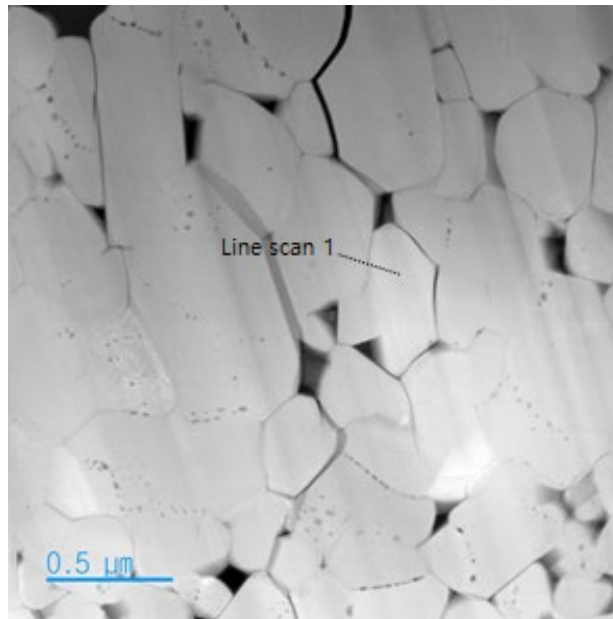


Figure J(1)

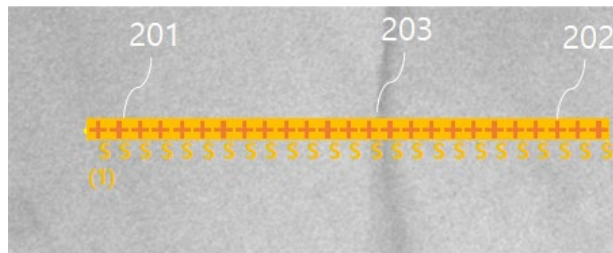


Figure J(2)

35. **Figure K(1)** represents a graph showing the concentration of Cobalt (as atomic %) from the STEM-EDS analysis performed at the locations shown in **Figure J(2)**.

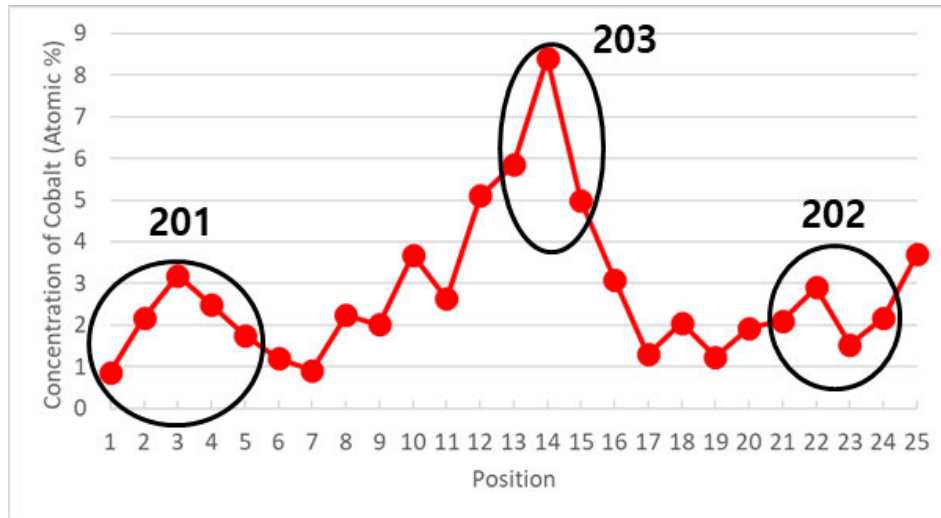


Figure K(1)

36. As seen in **Figure K(1)**, our experiment results confirmed that the concentration of Co in the areas marked as 201 and 202 in the bulk of the crystals is less than 4 atomic% and the Co concentration in the grain boundary between the crystals (spots marked as 203) was in the range of about 5 to about 9 atomic %.

37. For comparison, below is Figure 7 of the '317 patent, described as a graph of the concentration of cobalt versus position for each spot of the EDS analysis.

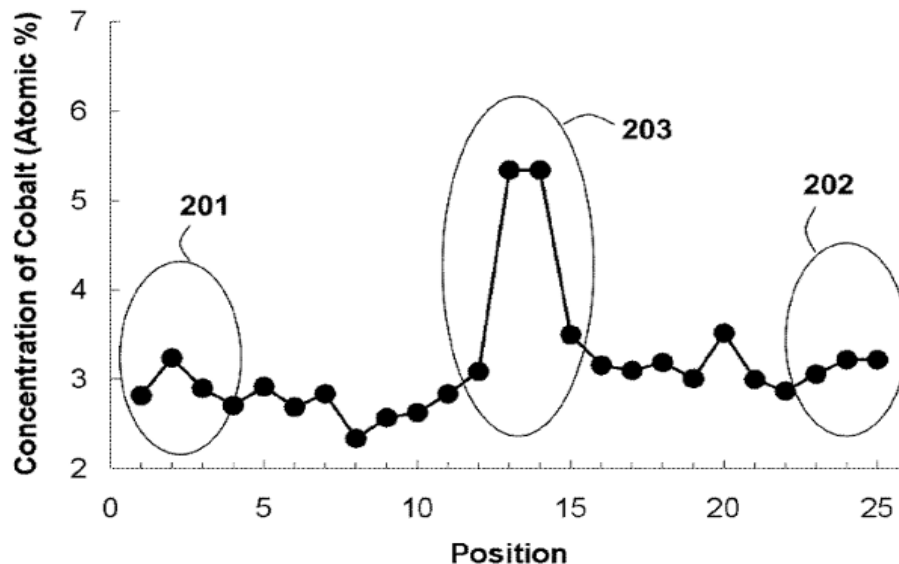


FIG. 7

III. Replication of Example 5 of Takagi

38. The purpose of this experiment is to perform the process in Example 5 of Takagi and then to analyze the shape and composition of the resulting particles. The manufacture of the particles was performed June 17, 2024 ~ June 19, 2024 at POSCO Future M's Gumi Cathode Material Plant. The characterization of the material was performed from June 19, 2024 to June 21, 2024 at GERI.

39. **Testing Method:** the left column corresponds to the steps disclosed in Example 5 of Takagi. The right column of the table below includes the testing method carried out under my supervision.

Example 5 of Takagi	Steps Taken Under My Supervision
Nickel hydroxide, lithium hydroxide, and cobalt hydroxide were mixed until a molar ratio reached 1:1:0.05	Step 1: The following metal hydroxide powders were mixed in a bottle (in a 1:1:0.05 molar ratio). ○ 226.59 g of LiOH H₂O ○ 499.96 g of Ni(OH)₂, ○ 25.2 g of Co(OH)₂
First firing step: contents fired in an oxygen stream at 200 °C for 3 hours.	Step 2: contents fired in oxygen stream at 200 °C for 3 hours.
Second firing step: contents fired in an oxygen stream at 500 °C for 5 hours.	Step 3: contents fired in an oxygen stream at 500 °C for 5 hours
Granulation step: mixing fired product with ion-exchange water at a mass ratio of 30:70, disaggregated and dispersed in wet bead mill for 1 hour, and granulated by spray granulation.	Step 4: the resulting material was then grinded in a wet bead mill for about 1 hour and then spray dried
Third firing step: contents fired in an oxygen stream at 700 °C for 5 hours.	Step 5: the contents were fired in an oxygen stream at 700 °C for 5 hours.

40. **Test Results:** After the final product was collected, it was analyzed using FIB, STEM and STEM-EDS.

41. Using the FIB, thin samples of about 80-100 nm thickness were produced by ion milling the final sample produced by the method listed above. The shape and structure of the thin samples were then analyzed using the STEM under 200 kV of acceleration voltage. The compositions

of the thin samples were then analyzed using STEM-EDS (EDS attached to the STEM).

42. **Figure L(1)** is a STEM image of a portion of the final product prepared under my supervision, as detailed above, for the replication of Example 5 of Takagi. **Figure L(2)** indicates an area that I selected for a line scan for cobalt concentration.

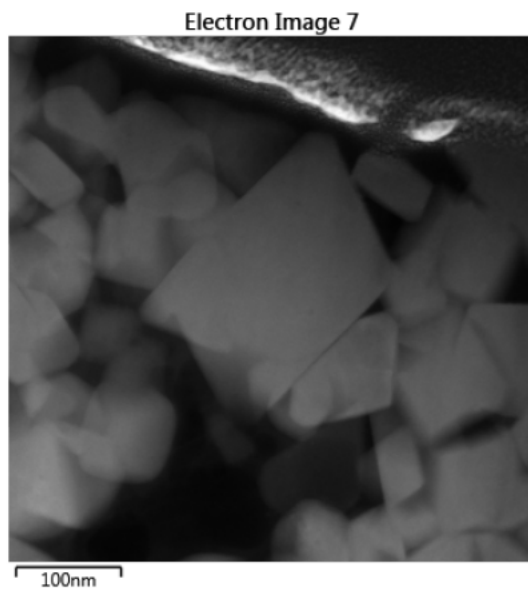


Figure L(1)

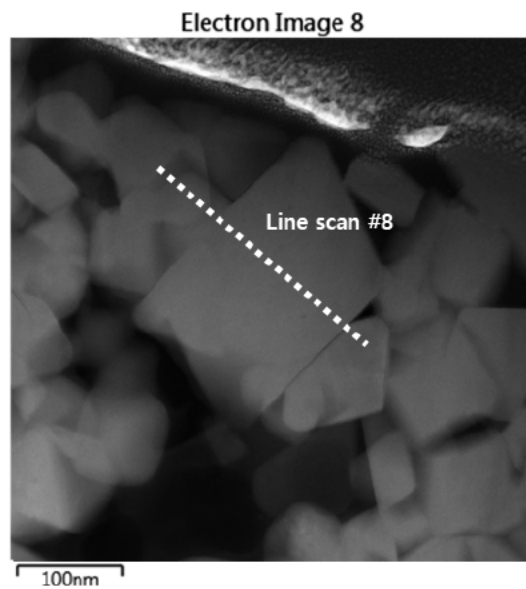


Figure L(2)

43. **Figure M(1)** depicts a close-up of 20 points where cobalt concentration is measured in line scan #8. **Figure M(2)** is a chart indicating the cobalt concentration by atomic percentage from STEM-EDS analysis performed at each position.

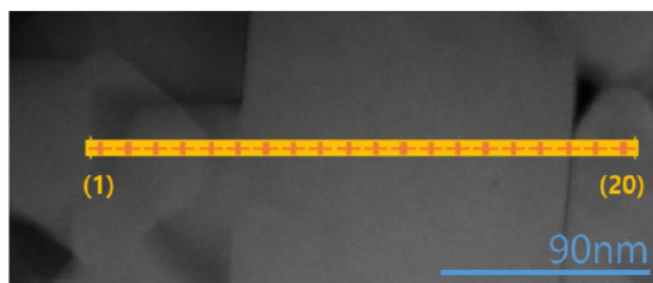


Figure M(1)

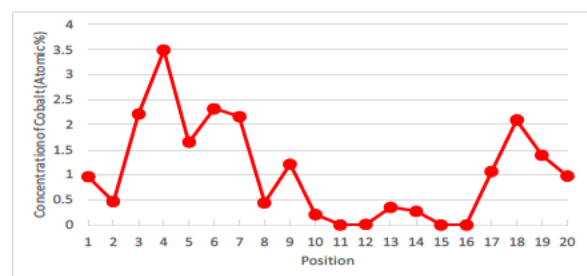


Figure M(2)

44. As seen in **Figure M(2)**, the experimental results confirmed that the concentration of Co in positions in the bulk of the crystals is less the concentration of Co in the grain boundary between the crystals. For example, in the bulk of the crystals, the atomic % is in the range of 0 to 0.5% at

positions 10-16, and 1% at position 20. In the grain boundaries, the atomic % is in the range of 2 to 2.5% at position 6-7, and 2% at position 18.

IV. Replication of Examples 1 and 7 of the Onnerud patent

45. According to the Onnerud patent, the purpose of Example 1 is to synthesize $\text{Li}_{1.05}\text{Mg}_{0.025}\text{NiO}_2$ composition and the purpose of Example 7 is to coat the synthesized $\text{Li}_{1.05}\text{Mg}_{0.025}\text{NiO}_2$ composition of Example 1 with a LiCoO_2 Layer.

46. **Testing Method:** The left column includes the steps disclosed in Examples 1 and 7 of the Onnerud patent. The right column of the table below includes the testing method conducted under my supervision. The weight of the precursors used in our testing method represent about 53% of the weight of the precursors used in Example 1 of the Onnerud patent and about 9% of the weight of the precursors used in Example 7 of the Onnerud patent. The manufacturing of specimens of Examples 1 and 7 were performed between April 22-23, 2024.

Example 1 of the Onnerud patent	Steps Taken Under My Supervision
A $\text{Li}_{1.05}\text{Mg}_{0.025}\text{NiO}_2$ composition was prepared by dry mixing: - o about 242.91 g $\text{Li}(\text{OH})_2^3$ - o about 14.79 g $\text{Mg}(\text{OH})_2$ - o about 34.97 g LiNO_3	Step 1: The following precursors were dry-mixed: - o 129.16 g of LiOH - o 7.86 g of $\text{Mg}(\text{OH})_2$, and - o 18.59 g of LiNO_3
The mixed materials were added to about 940.31 g $\text{Ni}(\text{OH})_2$ in a 1 liter jar. The compounds were mixed by shaking in the jar.	Step 2: The mixture of Step 1 was placed in a 1 L bottle containing 499.96 g of $\text{Ni}(\text{OH})_2$, then mixed by shaking.
The homogeneous precursor powders (precursor compounds) were placed in alumina crucibles and sintered.	Step 3: The mixture resulting from Step 2 was then placed into alumina crucible and sintered.
Sintering was performed by:	Step 4: The sintering included: - o heating at 5 °C/min to 450 °C, and - maintaining at 450 °C for 4 hours,

³ While the Onnerud patent discloses using $\text{Li}(\text{OH})_2$, we believe that this is a typographical error and, as such, we used LiOH in our experiment.

○ heating at a rate of about 5 °C/minute to about 450 °C and held at about 450 °C for about four hours. ○ The temperature was then raised at about 2 °C/minute to about 700 °C and held for about four hours The sample was then allowed to cool naturally to room temperature.	○ then heating at 2 °C/min from 450 °C to 700 °C and maintaining at 700 °C for 4 hours, and ○ then cooling to room temperature.
The cooled sample was ground for about five minutes to break up any agglomerates, and then the powder material was sieved through a No. 270 mesh to remove larger particles and to ensure a desired 10 µm particle size.	Step 5: The material of Step 4 was crushed for about 5 minutes and sieved through a No. 270 mesh.
Example 7 of the Onnerud patent: A lithium-magnesium-nickel oxide composition, substantially prepared as described in Example 1, was coated with a lithium-cobalt oxide layer.	
To synthesize the coating layer, the following materials were dissolved in about 200-300 ml distilled water: ○ about 105.55 g LiNO₃ and ○ about 445.50 g Co(NO₃)₂·6H₂O	Step 6: the following materials were dissolved in about 27 ml of distilled water: ○ 9.50 g of LiNO₃ and ○ 40.10 g of Co(NO₃)₂·6H₂O
About 1000 g of the Li_{1.05}Mg_{0.025}NiO₂ powder substantially prepared as described in Example 1 was added to the mixture.	Step 7: 90 g of the material prepared in Step 5 was added to the solution of Step 6 and mixed.
The excess water was removed by evaporation on a hot plate with stirring until the mixture became a thick slurry.	Step 8: The water in the solution in Step 7 was removed by evaporating on the hot plate until the mixture became thick slurry.
The slurry was poured into an alumina crucible and sintered under the following heating profile: ○ heat at a rate of about 2 °C/min to about 110 °C, hold for about one hour at about 110 °C, ○ heat at a rate of 5 °C./min to about 450 °C, heat soak for about one hour at about 450 °C, ○ heat at a rate of about 2 °C/min to about 700 °C, and heat soak for about two hours at about 700 °C.	Step 9: The thick slurry of Step 8 was then placed into alumina crucible and sintered as follows: ○ heating at 110 °C at 2 °C/min then maintaining at 110 °C for 1 hour, ○ then heating at 450 °C at 5 °C/min for 1 hour, ○ then heated at 450°C to 700°C at 2°C/min, maintained at 700°C for 2 hours.
The prepared sample was allowed to cool naturally to room temperature. Once cooled, it was ground for about five minutes to break up	Step 10: The sintered sample of Step 9 was then cooled to room temperature, then crushed for about 5 minutes, and then sieved

any agglomerates and sieved through a No. 270 mesh screen.

through a No. 270 mesh to produce the final product (“final product”).

47. **Test Results:** After the final product was collected, it was analyzed using FIB, STEM and STEM-EDS at the GERI laboratory between April 26-30, 2024.

48. Using the FIB, thin samples of about 80-100 nm thickness were produced by ion milling the final sample produced by the method listed above. The shape and structure of the thin samples were then analyzed using the STEM under 200 kV of acceleration voltage. The compositions of the thin samples were then analyzed using EDS built into the STEM. I attended the GERI laboratory when analyzing STEM and STEM-EDS and identified line scan locations for analysis of cobalt concentrations.

49. **Figure O** represents an STEM image of the final product prepared under my supervision.

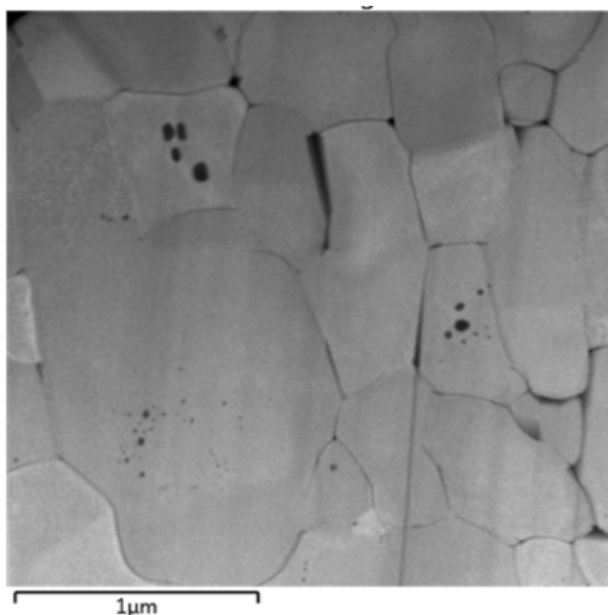


Figure O

50. **Figure P** represents an STEM-EDS elemental analysis of the same final product illustrated in **Figure O**. The areas shaded in red in **Figure P** represent cobalt concentration.

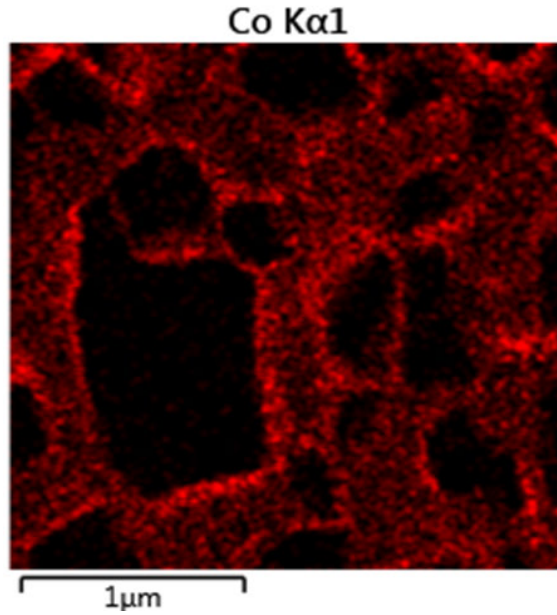


Figure P

51. **Figure Q(1)** is **Figure O** additionally marked with Line scan 3, showing a line scan I selected to measure the concentration of cobalt. **Figure Q(2)** shows 25 locations analyzed for measuring the concentration of Cobalt in Line scan 3 in **Figure Q(1)**, each marked as “S” in the yellow line.

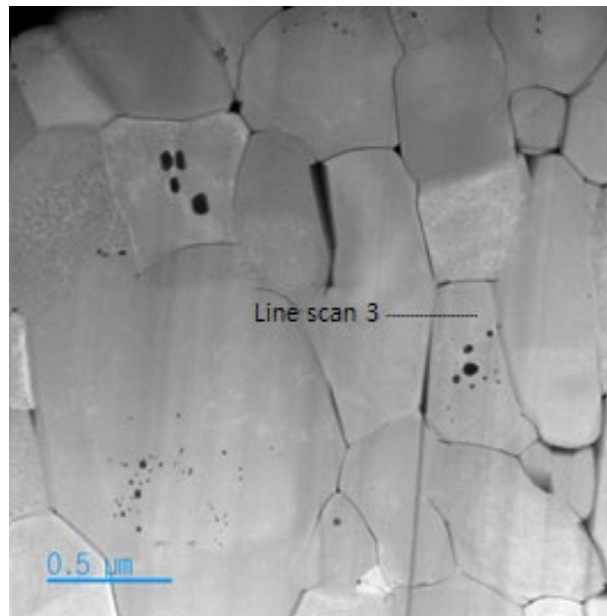


Figure Q(1)

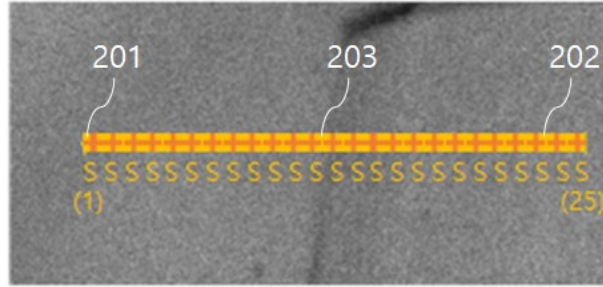


Figure Q(2)

52. Figure R represents a graph showing the concentration of Cobalt (as atomic %) from STEM-EDS analysis performed at the locations indicated in Figure Q(2).

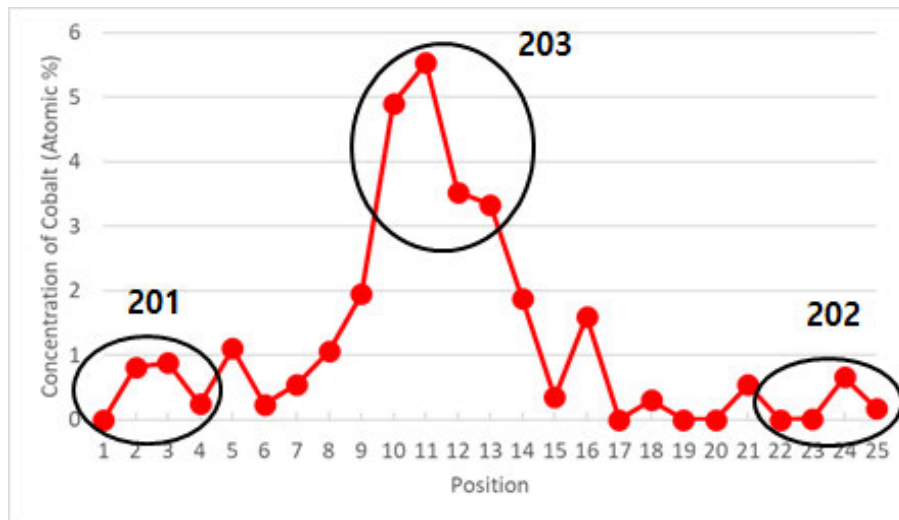


Figure R

53. As seen in **Figure R**, our experimental results confirmed that the concentration of Co in the areas marked as 201 and 202 in the bulk of the crystals was in the range of about 0 to less than 1, and the Co concentration in the grain boundary between the crystals (spot marked as 203) was in the range of about 3.5 to about 5.5 atomic %.

I declare, under penalty of perjury, that all statements made in this Declaration based on my own knowledge are true, and that all statements made based on information and belief are believed

by me to be true. I have been warned, and I understand, that willful false statements and the like are punishable by fine, imprisonment, or both (18 U.S.C. § 1001).

Date: July 23, 2024



Hyunkuk Noh, PhD