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(54) **Title:** CURRENT COLLECTOR



FIG. 1a



FIG. 1b

(57) **Abstract:** A method of making an assembly comprising a nickel current collector strained in the 111 crystallographic plane, and a hexagonal layer of LiCoO deposited thereupon. The method comprises: forming a nickel current collector by plasma sputter deposition, wherein the plasma bias power is $\geq 1.5\text{Kw}$ and the target bias power is $\geq 1.0\text{ kW}$; and using sputter deposition to form an electrode on the current collector.

CURRENT COLLECTOR

Technical Field

5 The present invention relates to a current collector and articles incorporating the same.

Background

10 Cathodes formed from LiCoO_2 are often employed in lithium ion batteries, and are typically disposed on current collectors. Lithium cobalt oxide forms a number of different crystalline structures and a hexagonal crystal form has been found to provide the best performance. However, significant energy input is required during LiCoO_2 deposition to form hexagonal crystals in prior art fabrication techniques. Moreover, antisite defects may be observed the lithium cobalt oxide structure, reducing performance. Such defects are removed in prior art techniques by calcination of the
15 LiCoO_2 material.

Summary

At its most general, the invention provides a current collector comprising a metal, wherein the metal is strained in a selected plane such that the lattice parameter
20 for the metal is within a selected range, and wherein the selected range of the lattice parameter provides control over the crystalline structure of an electrode material that may be deposited on the current collector. The inventors have established that control over strain in the current collector may be used to preferentially seed a particular crystal structure in a deposited electrode material.

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According to a first aspect of the present invention, there is provided a current collector comprising nickel, wherein the nickel is strained in the (111) crystallographic plane.

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A second aspect of the invention provides a component comprising a substrate and a current collector according to the first aspect, wherein the current collector is provided on the substrate.

A third aspect of the invention provides an assembly comprising a current collector according to the first aspect and an electrode.

5 A fourth aspect of the invention provides a battery comprising an assembly according to the third aspect.

A fifth aspect of the invention provides a method of making an assembly according to the third aspect, wherein the method comprises:

- 10 (i) forming a nickel current collector by plasma sputter deposition, wherein the plasma bias power is $\geq 1.5\text{Kw}$ and the target bias power is $\geq 1.0\text{ kW}$; and
- (ii) using sputter deposition to form an electrode on the current collector.

15 Further features and advantages of the invention will become apparent from the following description of preferred embodiments of the invention, given by way of example only, which is made with reference to the accompanying drawings.

To the extent that they are compatible, optional or preferable features of each aspect of the invention may be combined with other aspects of the invention defined
20 herein.

Brief Description of the Drawings

Figure 1a shows a schematic cross-sectional view of a current collector according to embodiments of the invention.

25 Figure 1b shows a schematic cross-sectional view of a component according to embodiments of the invention.

Figures 2a to 2e each show a schematic cross-sectional view of an assembly according to embodiments of the invention.

Figure 3 shows a schematic cross-sectional view of a battery according to
30 embodiments of the invention.

Figure 4 is a graph illustrating a relationship between (i) the nickel lattice parameter in a current collector, where nickel is strained in the 111 plane, and (ii) the c/a ratio of lattice parameters for a LiCoO₂ layer formed on the nickel current collector.

Figure 5 is a graph illustrating that there is no apparent relationship between (i) the nickel lattice parameter in a current collector, where nickel is strained in the 020 plane, and (ii) the c/a ratio of lattice parameters for a LiCoO₂ layer formed on the nickel current collector.

For reference, a c/a ratio for the LiCoO₂ layer of 5.0 represents a perfect hexagonal structure. A value of 4.90 is a more cubic lattice.

Detailed Description

Electrodes are typically formed on current collectors, and the inventors have established that that control over the direction of strain in the current collector may be used to preferentially seed a particular crystal structure in a deposited electrode material. In particular, for a nickel current collector, the inventors have established that strain the in 111 plane provides a current collector where there is a relationship between the degree of strain and the tendency of the current collector to seed a hexagonal crystalline form when LiCoO₂ is deposited on its surface. This is illustrated in Figure 4; it can be seen in Figure 4 that as the degree of lattice parameter for the 111-strained nickel increases, it preferentially seeds a more hexagonal crystalline form in the deposited LiCoO₂ structure.

Conversely, Figure 5 illustrates that there is no relationship between the degree of strain and the resulting seeded crystalline form where the nickel is strained in the 020 plane. There is no bias to a particular crystal form as the degree of strain changes.

For reference, un-strained nickel has a face-centred cubic lattice structure with a lattice parameter of 3.499 Å.

The lattice constant, or lattice parameter, refers to the physical dimension of unit cells in a crystal lattice and may be determined by x-ray diffraction. Lattices in three dimensions generally have three lattice constants, referred to as a, b, and c. However,

in the case of cubic crystal structures, all of the constants are equal and are referred to as a . Similarly, in hexagonal crystal structures, the a and b constants are equal, and typically reference is only made to the a and c constants.

5 The inventors have also determined that the level of antsite defects in the hexagonal LiCoO_2 formed on 111-strained nickel was less than 20%, whereas the level of defects observed with the 020-strained nickel was more than 20%. Maximum performance for the electrode is obtained by minimising the defect density. Thus, using the 111-strained nickel current collector reduces the defect level, and reduces the degree
10 of (or in some cases, obviates the need for) any subsequent defect-removal step (such as calcination). The required processing and required energy input needed to obtain an ideal electrode material is therefore reduced when depositing LiCoO_2 on nickel strained in the 111 plane.

15 Control over the directionality and degree of strain can therefore be used to preferentially seed a desired crystal structure, with a lower defect level, and can reduce the number of required processing steps and/or the energy input required in manufacture.

20 Control over strain may be achieved in a number of ways. For example, for a sputtering deposition, varying the impinging power density or the working distance during deposition can affect the strain. In a particular example, a conventional sputtering deposition can be used to produce highly strained nickel where the strain is the in 111 plane, by using high impinging power densities on the sputtering targets
25 ($> 5\text{W}/\text{cm}^2$) and or short working distances below the mean free path of the deposition process at lower target power densities.

 When manufacturing the current collectors which were evaluated in figures 4 and 5, a remote plasma sputtering process was employed similar to that described in
30 (Anguita, José & Thwaites, Michael & Holton, Barry & Hockley, Peter & Rand, Stuart & Haughton, Stuart. (2007). Room Temperature Growth of Indium-Tin Oxide on Organic Flexible Polymer Substrates Using a New Reactive-Sputter Deposition

Technology. Plasma Processes and Polymers. 4. 48 - 52. 10.1002/ppap.200600047). Highly strained nickel (strain in 111 plane) was deposited with a plasma bias power of ≥ 1.5 Kw and a target biasing power of ≥ 1.0 kW as sample-substrate separation approached the mean free path of the deposition system. Below these power thresholds
5 020-strained nickel was deposited.

In some cases, the nickel current collector, strained in the 111 plane, has a lattice parameter of greater than about 0.3516 nm, suitably greater than about 0.3517 nm, 0.3518 nm, 0.3519 nm, 0.3520 nm or 0.3521 nm. The inventors have established that
10 nickel strained in the 111 plane with a larger lattice parameter preferentially seeds a hexagonal crystalline form when LiCoO_2 is deposited on its surface.

In some cases, the current collector may be thin layer. In some cases, the thickness of the current collector may be less than about 100 μm , suitably less than
15 about 50 μm , less than about 10 μm , less than about 5 μm or possibly less than about 1 μm . Generally, a thin current collector is advantageous as it minimises the overall mass of the battery in which it employed. Typically, the current collector will be at least 500 nm thick, and possibly 1 μm or thicker. There is a lower limit on how thin the current collector can be before its mechanical strength and other physical properties
20 make it unsuitable for use.

In some cases, the current collector has a top surface which may support an electrode in use, wherein the surface roughness of the top surface is X_s , where $X_s \leq 100$ nm. The top surface of the current collector may have a surface roughness of
25 greater than 1 nm, for example greater than 5 nm.

It has been found that the roughness of the top surface of the current collector is an important factor in manufacturing products incorporating a thin layer of crystalline electrode material on the current collector. Ensuring that the surface of the current
30 collector is extremely smooth results in a crystalline electrode with surprisingly few defects. Conversely, when the surface of the current collector is relatively rough there is a surprisingly profound effect on the likelihood of delamination of the layer of

crystalline material from the current collector. If the top surface is too smooth however the current collector can become too difficult to handle and process.

Particularly in applications where the current collector is stored in layers that are in contact with each other, for example when the current collector is an elongate film supplied on a drum or roller, it may be that the surface roughness needed for easing handling decreases as the thickness of the current collector increases. For thick films of current collector, the minimum roughness required may be lower than for very thin films. It may be the case that the product of the thickness of the current collector and X_s (i.e. the thickness of the substrate multiplied by X_s) is no more than 10^5 nm^2 . Preferably, the product of the thickness of the current collector and X_s is no more than $5 \times 10^4 \text{ nm}^2$. In some cases, it may be that X_s is no more than 10 % of the thickness of the current collector.

The surface roughness may be measured by a profilometer. The surface roughness may be measured by means of calculating the RMS roughness. The RMS roughness may be calculated as the deviation in height from a perfectly smooth external surface. It will be understood that a perfectly smooth external surface is perfectly flat when the mid-plane of the substrate is transformed onto a flat plane. The surface roughness may be measured by means of calculating the arithmetic average of the absolute values of profile heights (above the minimum height measured) over an evaluation length of a sample.

Figure 1a shows a current collector 104. In an embodiment, the current collector is nickel strained in the 111 plane, but other materials may be used as discussed herein.

In some cases, the current collector may be provided as part of a component, wherein the current collector is provided on a substrate (and suitably is abutting the substrate). In some cases, the substrate may be flexible or rigid. In some cases, the substrate comprises one or more materials selected from: a polymer material, a semiconductor wafer, plastic film, metal foil, thin glass, mica and a polyimide material. In some cases, the substrate may comprise polyethylene terephthalate (PET), or

polyethylene naphthalate (PEN). PEN and PET are reasonably flexible, and have a relatively high tensile strength due to their semi-crystalline structure.

5 In some cases, the substrate may have a thickness of less than about 100 μm , suitably less than about 50 μm , and optionally more than about 0.5 μm , more than about 1 μm , or possibly more than about 10 μm .

10 In some cases, the substrate may be a thin film of material. Thus, the component may have a laminate structure, with a thin-layer current collector supported on a thin-layer substrate.

In some cases, the component may comprise, or be in the form of, a sheet, optionally an elongate sheet. Such a sheet may be provided in the form of a roll. This facilitates simple storage and handling of the component.

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Figure 1b shows a component 100 comprising a substrate layer 102 and a current collector layer 104 disposed on the substrate layer 102. In an embodiment, the substrate is formed from PET and the current collector is nickel strained in the 111 plane, but other materials may be used as discussed herein.

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In some cases, the invention provides an assembly comprising an electrode and the current collector described herein. In some cases, the electrode and current collector may be thin layers, where the electrode layer is provided on the current collector layer (and suitably abutting the current collector layer). In some cases, the assembly may additionally comprise a substrate, which is provided on the opposite side of the collector from the electrode. The assembly may comprise a laminate structure comprising a substrate layer, a current collector layer on (e.g. abutting) the substrate layer, and an electrode layer on (e.g. abutting) the current collector layer. In some cases, there may be intermediate layers between the layers recited above. In other cases, the recited layers may be in a directly abutting relationship.

30

In some cases, the electrode is a cathode and comprises LiCoO_2 . In some such cases, the thickness of the cathode is less than about $10\text{ }\mu\text{m}$. In some cases, the thickness of the cathode is from about 50 nm , 100 nm or 500 nm to about $1\text{ }\mu\text{m}$, $5\text{ }\mu\text{m}$ or $10\text{ }\mu\text{m}$.

5 At least a portion of and optionally all the electrode material may have a crystalline “layered oxide” structure. Such “layered oxide” structures are important when manufacturing solid-state batteries. A layered oxide structure allows for lithium ions to more easily de-intercalate from the crystal structure, resulting in a faster charging, higher capacity solid-state battery. It will be understood that intercalation
10 refers to a property of a material that allows ions to readily move in and out of the material without the material changing its phase (chemical and crystalline structure). For example, a solid-state intercalation film remains in a solid state during discharging and charging of an energy-storage device.

15 As noted above, for a cathode comprising LiCoO_2 , the material is crystalline and preferably has a hexagonal and/or rhombohedral lattice structure, optionally having a form which is in the $R\bar{3}m$ space group (also referred to as the “ $R\ 3(\text{bar})\ 2/m$ ” space group or space group 166). This structure has a number of benefits, such as having a relatively greater accessible capacity and high rate of charging and discharging
20 compared to the low energy structure of LiCoO_2 , which has a structure in the $Fd\bar{3}m$ space group (a face centred cubic structure). The $R\bar{3}m$ space group is regarded as having better performance in typical battery applications due to enhanced reversibility and fewer structural changes on lithium intercalation and de-intercalation. Therefore, crystalline LiCoO_2 in the $R\bar{3}m$ space group is favoured for solid state battery
25 applications.

 In some cases, the ratio of the lattice constants c/a for the LiCoO_2 cathode material is greater than about 4.90, suitably greater than about 4.92 or 4.96. (A c/a value of 5.0 represents a perfect hexagonal structure. A value of 4.90 is more cubic.)
30

 In some cases, the crystallite size of the LiCoO_2 cathode is in the range of about 10 nm to about 30 nm , suitably in the range of about 14 nm to about 25 nm .

In some cases, it may be that X_s (the surface roughness of the current collector top surface) is no more than 10 % of the thickness of the first electrode crystalline layer deposited on the current collector. For very thin electrode crystalline layers, from say
5 10nm to 100nm thick, it may be that X_s is no more than 50% of the thickness of the first electrode crystalline layer. For electrode crystalline layers, with a thickness of from 100nm to 1 μ m thick, it may be that X_s is no more than 10 % of the thickness of the first electrode crystalline layer.

10 In some cases, the LiCoO_2 cathode material may be deposited on the current collector by conventional sputtering or a variant of sputtering. It has been observed that higher energy depositions of LiCoO_2 favour the formation of hexagonal lattices. As noted above, prior art techniques for generating hexagonal LiCoO_2 typically involved heating the material (to $>500^\circ\text{C}$, i.e. calcination) to encourage hexagonal crystallisation.

15 When using a 111-strained nickel current collector as sputtering target, the power density required to generate a hexagonal LiCoO_2 cathode structure is lower. In some cases, a remote plasma sputtering process may be employed to deposit a LiCoO_2 layer, where the process is the similar to that described in (Anguita, José & Thwaites, Michael & Holton, Barry & Hockley, Peter & Rand, Stuart & Haughton, Stuart. (2007).
20 Room Temperature Growth of Indium-Tin Oxide on Organic Flexible Polymer Substrates Using a New Reactive-Sputter Deposition Technology. Plasma Processes and Polymers. 4. 48 - 52. 10.1002/ppap.200600047). The inventors have found that crystalline LiCoO_2 with a hexagonal structure could be deposited on 111-strained nickel
25 with a plasma bias power of $\geq 1.2\text{Kw}$ and a target biasing power of $\geq 0.6\text{ kW}$ as sample-substrate separation approached the mean free path of the deposition system. Importantly, there was no need for additional heating (i.e. calcination) to form hexagonal crystalline forms. Below these power thresholds a cubic/spinel phase of LiCoO_2 was formed.

30 As noted above, the invention also provides a method of making an assembly as described herein, wherein the method comprises:

- (i) forming a nickel current collector by plasma sputter deposition, wherein the plasma bias power is ≥ 1.5 Kw and the target bias power is ≥ 1.0 kW; and
- (ii) using sputter deposition to form an electrode on the current collector.

5 These sputtering conditions in step (i) result in the formation of a 111-strained nickel current collector, allowing lower energy deposition of the electrode material in step (ii), whilst still forming the required electrode crystal form.

10 In some cases, the electrode is a cathode formed from LiCoO_2 using plasma sputter deposition, wherein the plasma bias power is ≥ 1.2 Kw and the target bias power is ≥ 0.6 kW. The inventors have found that a hexagonal crystalline lithium cobalt oxide cathode can be formed at lower deposition energies as a result of using a 111-strained nickel target (which preferentially seeds the desired hexagonal form). In some particular cases, no additional heating step is included; the hexagonal crystal form is
15 generated without additional heating (e.g. calcination).

 In some cases, the electrode is a cathode formed from LiCoO_2 , and the impinging power density when forming the cathode by sputter deposition is from about XX to about YY, suitably from about XX to about ZZ.

20

 Figure 2a shows an assembly 200 according to an embodiment of the invention, comprising a current collector layer 104 and an electrode layer 106 disposed on the current collector layer 104. Figure 2b shows an assembly 210 according to another embodiment of the invention, comprising a component 100, comprising a substrate
25 layer 102 and a current collector layer 104 disposed on the substrate, and an electrode layer 106 disposed on the current collector layer 104 on the opposite side from the substrate layer 102. In some of these embodiments the substrate layer 102 (if present) is formed from PET, the current collector layer 104 is formed from nickel strained in the 111 plane and the electrode layer 106 is a LiCoO_2 cathode, but other materials may
30 be used as discussed herein.

In some cases, the assembly further comprises an electrolyte on the opposite side of the electrode from the current collector. In some such cases, the assembly may comprise a laminate structure comprising the following layers, in order: (a) a current collector layer, (b) a cathode layer, and (c) an electrolyte layer. In some cases, there may be intermediate layers between the layers recited above. In other cases, the recited layers may be in a directly abutting relationship.

Suitably, the assembly may comprise a laminate structure comprising the following layers, in order: (i) a substrate layer, (ii) a current collector layer, (iii) a cathode layer, and (iv) an electrolyte layer. In some cases, there may be intermediate layers between the layers recited above. In other cases, the recited layers may be in a directly abutting relationship. An example of such an assembly 220 is illustrated schematically in Figure 2c, in which the electrolyte layer 108 is disposed on a surface of the cathode layer 106 opposite to the current collector 104. The electrolyte layer may be LiPON, but other materials can be employed, as discussed herein. (Figure 2c employs the same reference numerals as earlier figures and description of these elements is provided in detail above.)

The electrolyte may comprise a solid layer, and may be referred to as a fast ion conductor. A solid electrolyte layer may have structure which is intermediate between that of a liquid electrolyte, which for example lacks a regular structure and includes ions which may move freely, and that of a crystalline solid. A crystalline material for example has a regular structure, with an ordered arrangement of atoms, which may be arranged as a two-dimensional or three-dimensional lattice. Ions of a crystalline material are typically immobile and may therefore be unable to move freely throughout the material.

In some cases, the electrolyte may have a thickness in the range of about 0.1 μm to about 10 μm . The electrolyte may comprise, or optionally be formed of, any suitable material which is ionically conductive, but which is also an electrical insulator, such as lithium phosphorous oxynitride (LiPON).

In some cases, the assembly further comprises a second electrode on the opposite side of the electrolyte from the first electrode. In some such cases, the second electrode is an anode, and may suitably comprise, or optionally be formed of, graphite, silicon and/or indium tin oxide. In some such cases, the assembly may comprise a laminate structure comprising the following layers, in order: (i) an optional substrate layer, (ii) a current collector layer, (iii) a cathode layer, (iv) an electrolyte layer, and (v) an anode layer. In some cases, there may be intermediate layers between the layers recited above. In other cases, the recited layers may be in a directly abutting relationship. An example of such an assembly is illustrated schematically in Figure 2d, in which the anode layer 110 is disposed on the opposite surface of the electrolyte layer 106 from the cathode layer 104. The anode layer may be graphite, but other materials can be employed, as discussed herein. (Figure 2d employs the same reference numerals as earlier figures and description of these elements is provided in detail above.)

Although not illustrated, any assembly with an anode layer may include a current collector associated with the anode. This may be a further layer of a metal, for example, nickel, disposed on a surface of the anode opposite to the electrolyte.

A further assembly 250 is illustrated in Figure 2e. In this assembly, a laminate structure consisting of the following abutting layers, in order is provided: a substrate 102, suitably formed from PET; a current collector 104, suitably formed from 111-strained nickel; a cathode layer 106, suitably formed from LiCoO_2 ; an electrolyte layer 108, suitably formed from LiPON; an anode layer 110, suitably formed from graphite; a second electrolyte layer 108', suitably formed from LiPON; a second cathode layer 106', suitably formed from LiCoO_2 ; and a second current collector layer 104', suitably formed from 111-strained nickel. Although not illustrated, any assembly with an anode layer may include a current collector associated with the anode. This may be a further layer of a metal, for example, nickel, disposed within the anode (e.g. bisecting the anode, in a plane parallel to the other layers).

30

Further, as will be envisaged, in further embodiments, the assembly may comprise further layers stacked above 104', repeating the order shown in Figure 2e.

In all cases described herein, the current collector, component or assembly may be flexible, such that it can be rolled and processed on roll-to-roll apparatus (also known as reel-to-reel apparatus).

5

A further aspect of the invention provides a battery comprising a least one assembly described herein. In some cases, the battery comprises a plurality of assemblies, suitably a plurality of assemblies. Such a battery 300 is illustrated in Figure 3, where two assemblies 240a and 240b are illustrated. (The reference numerals from previous figures are used again in Figure 3. The two assemblies and constituent elements have been denoted as “a” and “b” suffixes on the reference numerals.) An electrically insulating material 302 is arranged between the assemblies. The electrically insulating material 302 may be an ink, such as a dielectric ink. A suitable dielectric ink is DM-INI-7003, available from Dycotec Materials Ltd., Unit 12 Star West, Westmead Industrial Estate, Westlea, Swindon, SN5 7SW, United Kingdom. In general, the electrically insulating material 302 may be any suitable dielectric material. A dielectric material is for example an electrical insulator which may be polarized upon application of an electric field. Such a dielectric material typically also has a low electrical conductivity.

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In the battery of Figure 3, two assemblies are illustrated. In other, non-illustrated embodiments, there may be a single assembly, and in other embodiments, there may be more than two assemblies.

25

Throughout this specification, reference to an element being “on” another element is to be understood as including direct or indirect contact. In other words, an element on another element may be either touching the other element, or not in contact the other element but, instead, generally supported by an intervening element (or elements) but nevertheless located above, or overlapping, the other element. In some cases, the elements are adjacent i.e. in direct contact or abutting. In some cases, the materials may adhere to one another. Where there are a number of stacked materials

30

(such as in the laminate structures described herein), some of the layers may adhere to one another but others need not adhere.

The above embodiments are to be understood as illustrative examples of the invention. It is to be understood that any feature described in relation to any one embodiment may be used alone, or in combination with other features described, and may also be used in combination with one or more features of any other of the embodiments, or any combination of any other of the embodiments. Furthermore, equivalents and modifications not described above may also be employed without departing from the scope of the invention, which is defined in the accompanying claims.

CLAIMS

1. A current collector comprising nickel, wherein the nickel is strained in the 111 crystallographic plane.
- 5 2. A current collector according to claim 1, wherein the lattice parameter of the nickel is greater than about 0.3516 nm.
- 10 3. A current collector according to claim 2, wherein the lattice parameter of the nickel is greater than about 0.3521 nm.
4. A current collector according to any preceding claim, wherein the thickness of the current collector is less than about 100 μm .
- 15 5. A current collector according to any preceding claim, wherein the current collector has a top surface which may support an electrode in use, wherein the surface roughness of the top surface is X_s , where $X_s \leq 100 \text{ nm}$.
- 20 6. A component comprising a substrate and a current collector according to any preceding claim, wherein the current collector is provided on the substrate.
- 25 7. A component according to claim 6, wherein the substrate comprises one or more materials selected from: a polymer material, a semiconductor wafer, plastic film, metal foil, thin glass, mica and a polyimide material.
8. A component according to claim 6 or claim 7, wherein the substrate has a thickness of from 0.5 μm to 100 μm .
- 30 9. An assembly comprising a current collector according to any one of claims 1 to 5 and an electrode.
10. An assembly according to claim 9, wherein the electrode is a cathode which comprises LiCoO_2 .

11. An assembly according to claim 10, wherein the thickness of the cathode is less than about 10 μm .
- 5 12. An assembly according claim 10 or claim 11, wherein crystallite size of the cathode is in the range of 10 nm to 30 nm.
- 10 13. An assembly according to any of claims 9 to 12, further comprising a substrate, wherein the substrate is provided on the opposite side of the current collector from the electrode.
14. An assembly according to any of claims 9 to 13, further comprising an electrolyte on the opposite side of the electrode from the current collector.
- 15 15. An assembly according to claim 14, further comprising a second electrode on the opposite side of the electrolyte from the first electrode.
16. An assembly according to claim 15, wherein the second electrode is an anode which comprises graphite.
- 20 17. A battery comprising an assembly according to any one of claims 9 to 16.
18. A battery according to claim 17, where the battery comprises a plurality of assemblies according to any one of claims 9 to 16.
- 25 19. A method of making an assembly according to any one of claims 9 to 16, wherein the method comprises:
- 30 (i) forming a nickel current collector by plasma sputter deposition, wherein the plasma bias power is $\geq 1.5\text{Kw}$ and the target bias power is $\geq 1.0\text{ kW}$; and
- (ii) using sputter deposition to form an electrode on the current collector.
- 35 20. A method according to claim 19, wherein in step (ii) the electrode is a cathode formed from LiCoO_2 as is formed using plasma sputter deposition,

wherein the plasma bias power is $\geq 1.2\text{Kw}$ and the target bias power is $\geq 0.6\text{ kW}$.

- 5 21. A method according to claim 20, wherein the LiCoO_2 has a hexagonal crystalline form and wherein the method does not include any additional heating step to generate this crystal form.



FIG. 1a

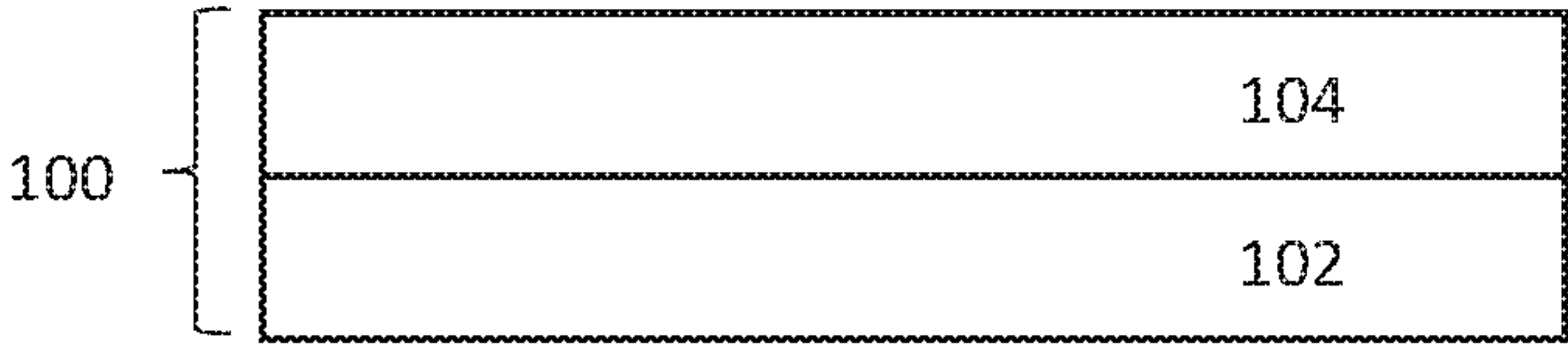


FIG. 1b



FIG. 2a

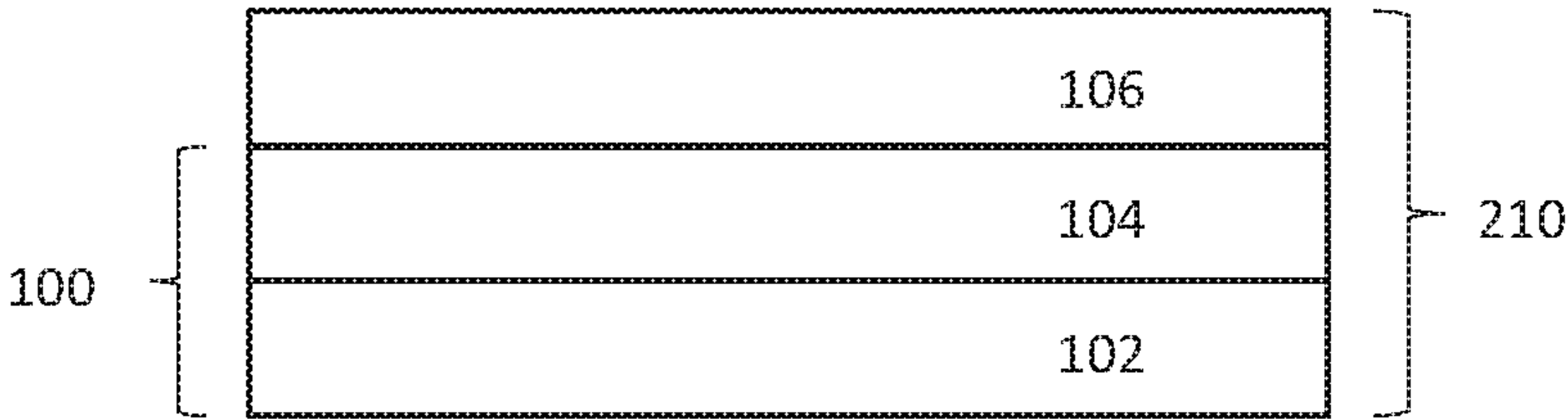


FIG. 2b

2 / 4

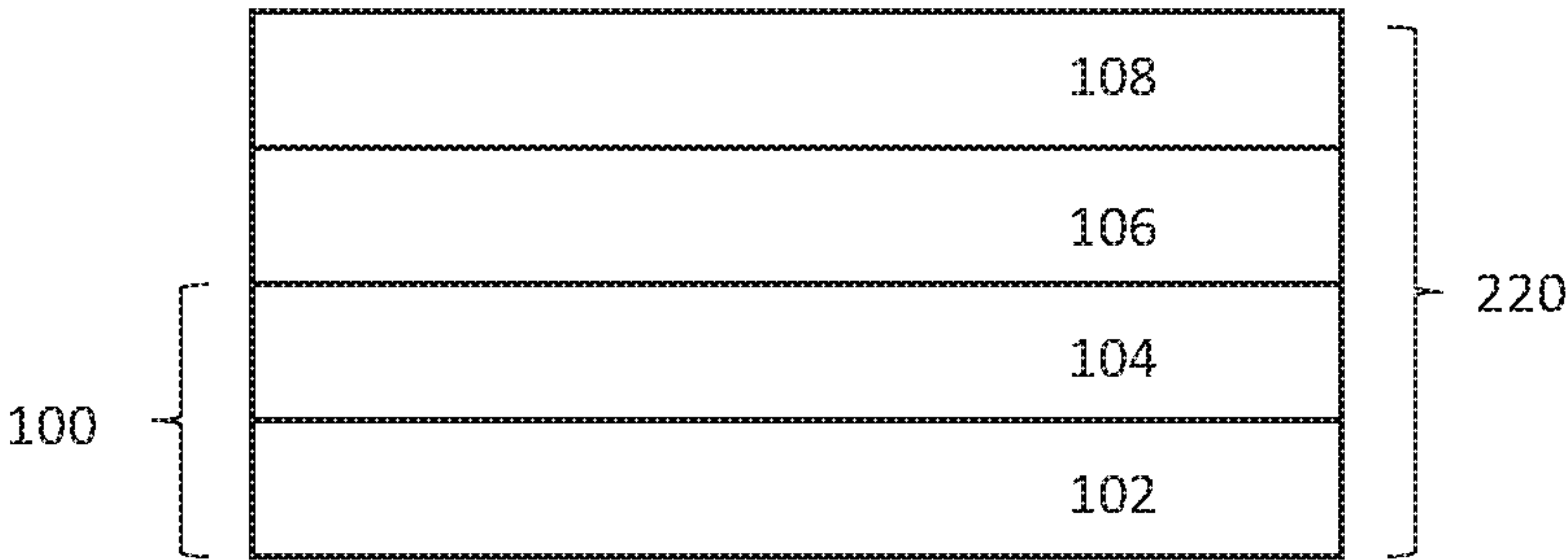


FIG. 2c

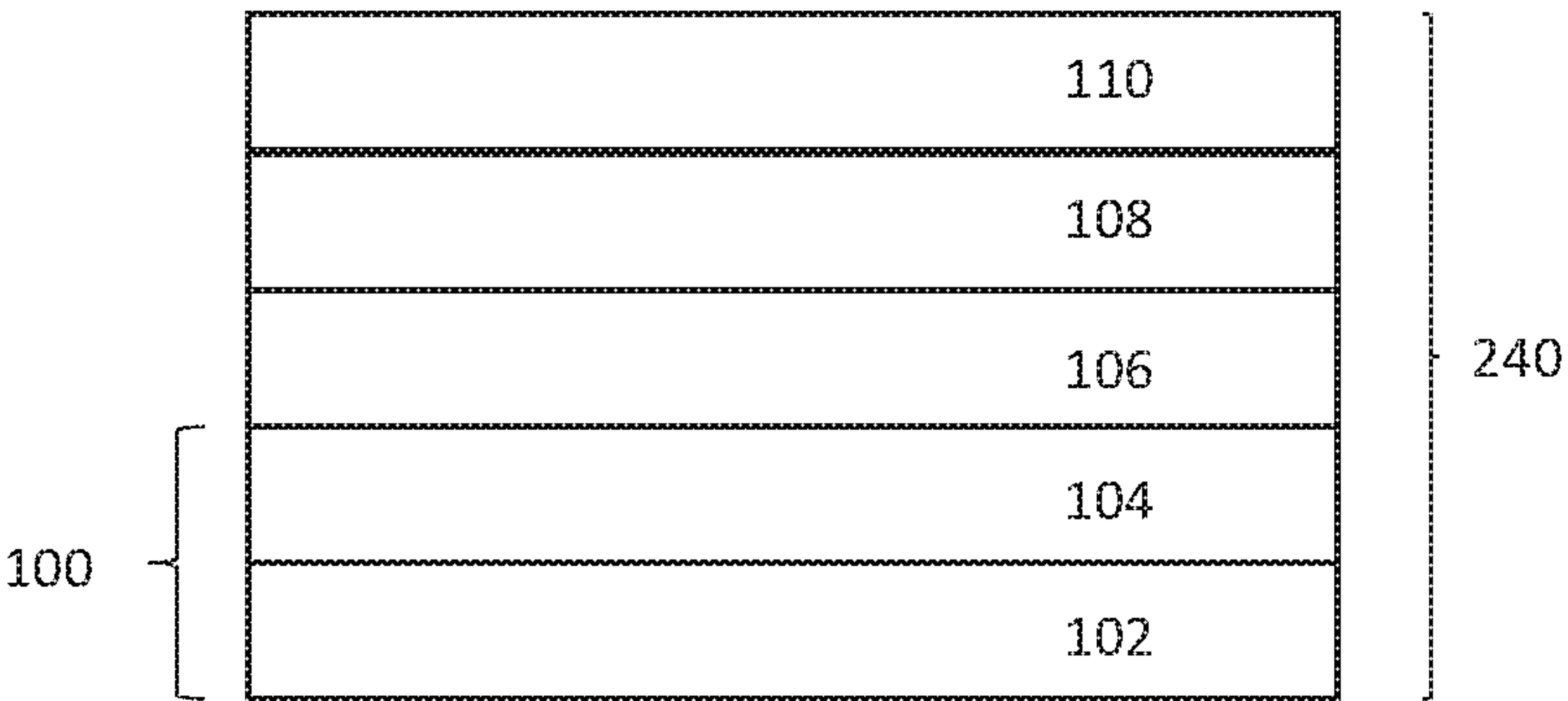


FIG. 2d

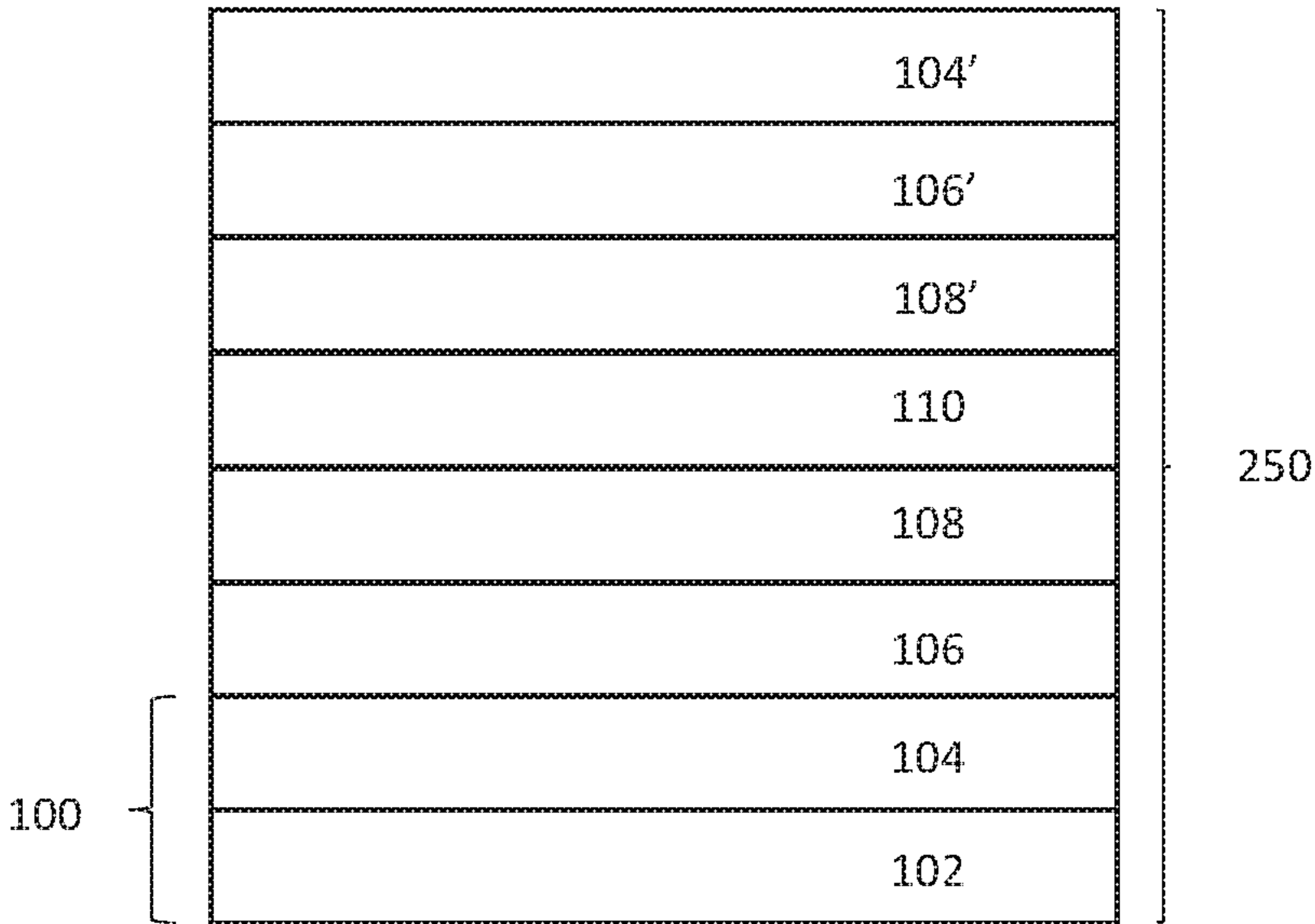


FIG. 2e

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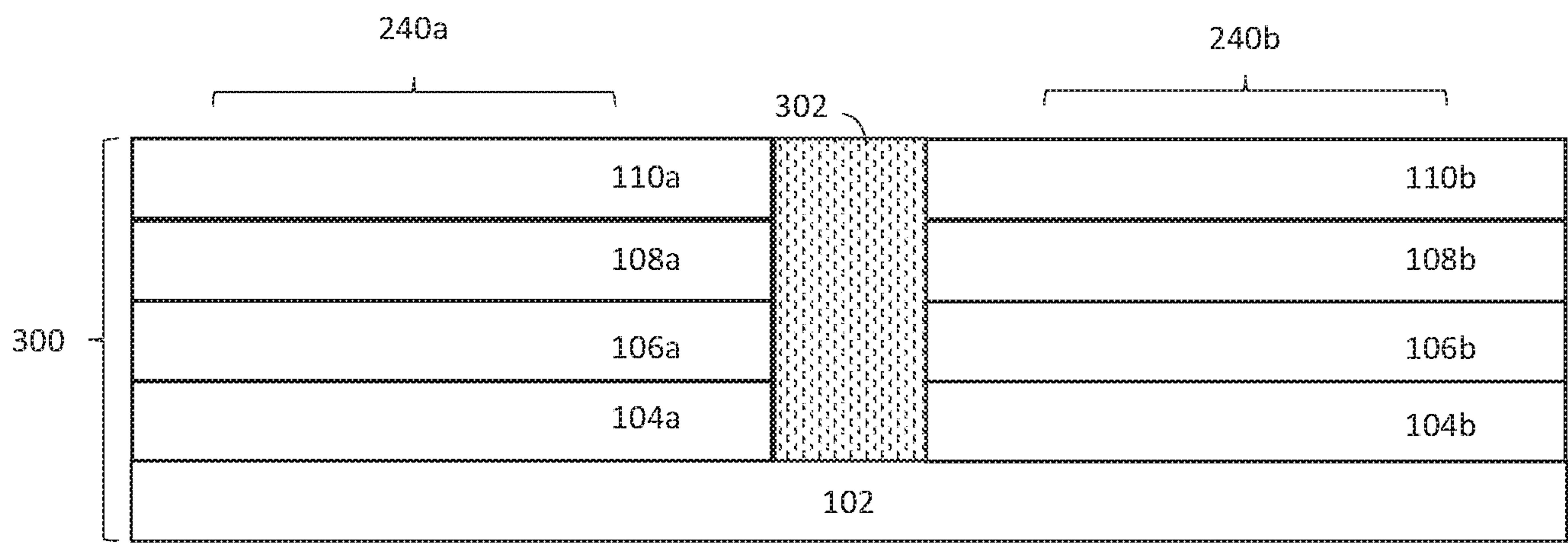


FIG. 3

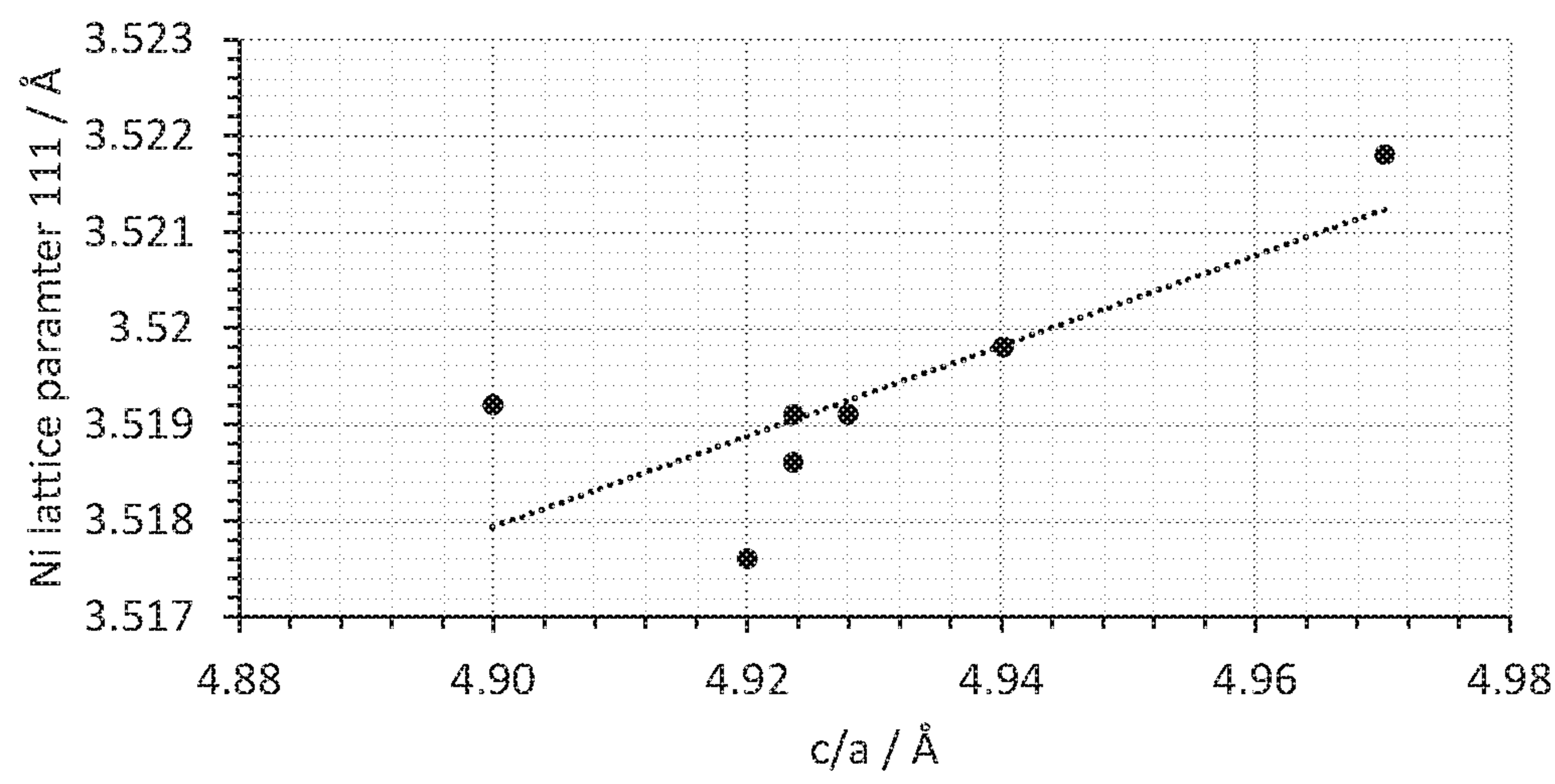


FIG. 4

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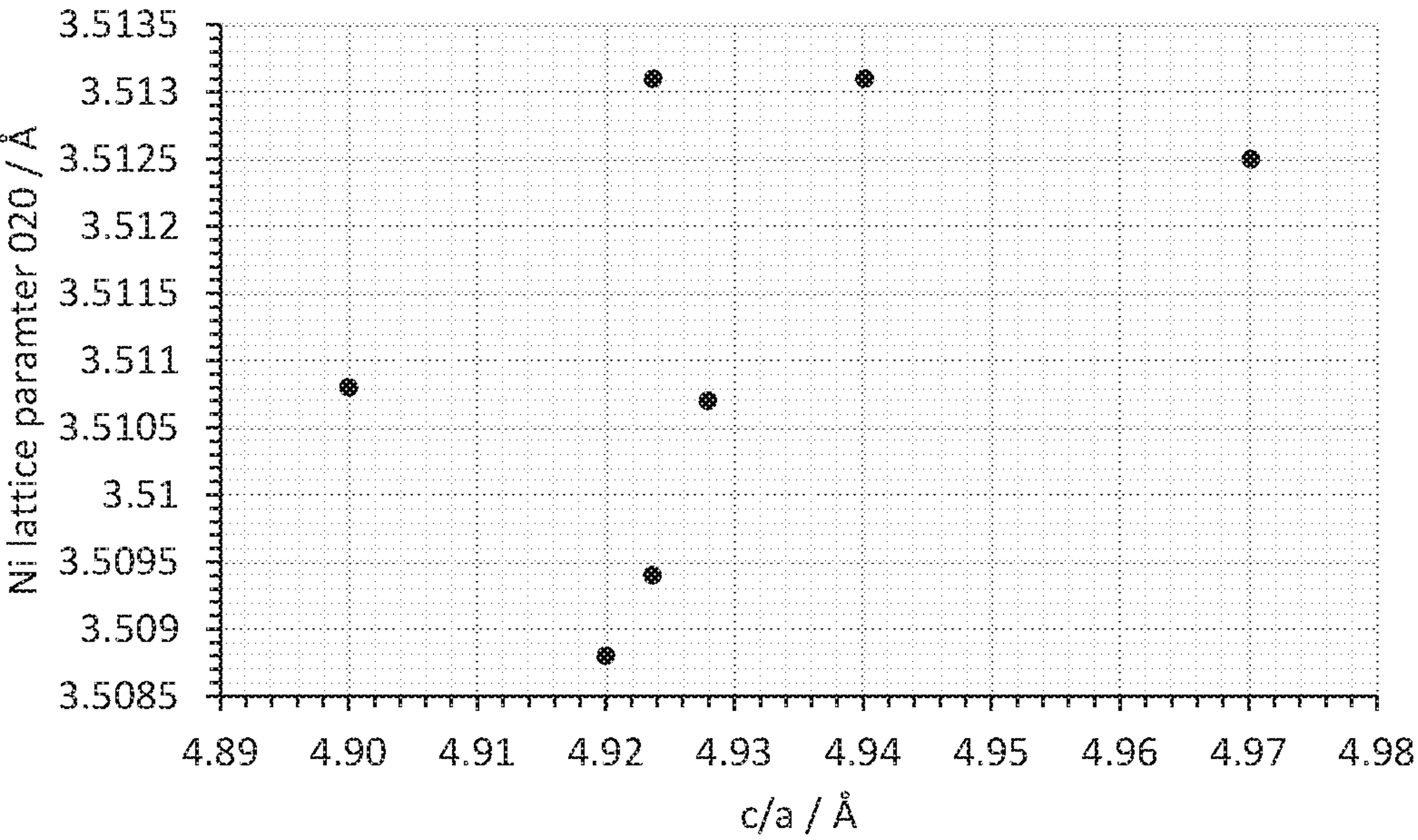


FIG. 5

INTERNATIONAL SEARCH REPORT

International application No

PCT/GB2020/053118

A. CLASSIFICATION OF SUBJECT MATTER
INV. H01M4/64 H01M4/66
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
H01M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	ANGUITA JOSE V. ET AL: "Room Temperature Growth of Indium-Tin Oxide on Organic Flexible Polymer Substrates Using a New Reactive-Sputter Deposition Technology", PLASMA PROCESSES AND POLYMERS, vol. 4, no. 1, 5 January 2007 (2007-01-05), pages 48-52, XP055774952, DE ISSN: 1612-8850, DOI: 10.1002/ppap.200600047 cited in the application the whole document ----- -/--	1-21



Further documents are listed in the continuation of Box C.



See patent family annex.

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Date of the actual completion of the international search

11 February 2021

Date of mailing of the international search report

22/02/2021

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Radeck, Stephanie

INTERNATIONAL SEARCH REPORT

International application No
PCT/GB2020/053118

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
T	Tran Denh: "High target utilisation sputtering for the development of advanced materials for magnetic data storage applications", 1 January 2012 (2012-01-01), pages 1-233, XP055775224, Exeter Retrieved from the Internet: URL:https://ore.exeter.ac.uk/repository/bitstream/handle/10871/8001/TranD.pdf?sequence=2&isAllowed=y page 28 - page 34 -----	
A	BYUN SEGI ET AL: "Direct formation of a current collector layer on a partially reduced graphite oxide film using sputter-assisted metal deposition to fabricate high-power micro-supercapacitor electrodes", JOURNAL OF POWER SOURCES, ELSEVIER SA, CH, vol. 307, 1 February 2016 (2016-02-01), pages 849-855, XP029410427, ISSN: 0378-7753, DOI: 10.1016/J.JPOWSOUR.2016.01.054 the whole document -----	1-21