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APPLICATION NUMBER	FILING or 371(c) DATE	GRP ART UNIT	FIL FEE REC'D	ATTY. DOCKET NO.	TOT CLAIMS	IND CLAIMS
61/575,115	08/16/2011		110			

TIAX LLC
c/o Nadine Andon
35 Hartwell Avenue
Lexington, MA 02421-3102

CONFIRMATION NO. 4911
FILING RECEIPT



Date Mailed: 08/26/2011

Receipt is acknowledged of this provisional patent application. It will not be examined for patentability and will become abandoned not later than twelve months after its filing date. Any correspondence concerning the application must include the following identification information: the U.S. APPLICATION NUMBER, FILING DATE, NAME OF APPLICANT, and TITLE OF INVENTION. Fees transmitted by check or draft are subject to collection. Please verify the accuracy of the data presented on this receipt. **If an error is noted on this Filing Receipt, please submit a written request for a Filing Receipt Correction. Please provide a copy of this Filing Receipt with the changes noted thereon. If you received a "Notice to File Missing Parts" for this application, please submit any corrections to this Filing Receipt with your reply to the Notice. When the USPTO processes the reply to the Notice, the USPTO will generate another Filing Receipt incorporating the requested corrections**

Applicant(s)

David Ofer, Needham, MA;
Adrian Wesley Pullen, Jamaica Plain, MA;

Power of Attorney: None

If Required, Foreign Filing License Granted: 08/24/2011

The country code and number of your priority application, to be used for filing abroad under the Paris Convention, is **US 61/575,115**

Projected Publication Date: None, application is not eligible for pre-grant publication

Non-Publication Request: No

Early Publication Request: No

**** SMALL ENTITY ****

Title

Cobalt enrichment at grain boundaries of polycrystalline layered metal oxides

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Title 35, United States Code, Section 184

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U.S. PTO
61/575115
08/16/2011

PTO/SB/16 (12-08)

Approved for use through 06/30/2010. OMB 0651-0032

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PROVISIONAL APPLICATION FOR PATENT COVER SHEET – Page 1 of 2

This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(c).

Express Mail Label No. EO 052 288 914 US

081611

INVENTOR(S)		
Given Name (first and middle (if any))	Family Name or Surname	Residence (City and either State or Foreign Country)
David	Ofer	Needham, MA
Adrian Wesley	Pullen	Jamaica Plain, MA

Additional inventors are being named on the n/a separately numbered sheets attached hereto.

TITLE OF THE INVENTION (500 characters max):

COBALT ENRICHMENT AT GRAIN BOUNDARIES OF POLYCRYSTALLINE LAYERED METAL OXIDES

Direct all correspondence to: **CORRESPONDENCE ADDRESS**

☐ The address corresponding to Customer Number:

OR

☒ Firm or Individual Name **TIAX LLC % Nadine Andon**

Address **35 Hartwell Avenue**

City Lexington	State MA	Zip 02421-3102
Country USA	Telephone 781/879-1283	Email andon.n@tiaxllc.com

ENCLOSED APPLICATION PARTS (check all that apply)

☒ Application Data Sheet. See 37 CFR 1.76 ☐ CD(s), Number of CDs _____

☐ Drawing(s) Number of Sheets _____ ☐ Other (specify) _____

☒ Specification (e.g. description of the invention) Number of Pages 6

Fees Due: Filing Fee of \$220 (\$110 for small entity). If the specification and drawings exceed 100 sheets of paper, an application size fee is also due, which is \$270 (\$135 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s).

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☒ Applicant claims small entity status. See 37 CFR 1.27.

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\$110.00
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OSCO Future M. TIAX LLC
OSCO Future M EX1006

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SIGNATURE

Joseph M. Centofanti

Date AUG. 16, 2011

TYPED or PRINTED NAME

Joseph M. Centofanti

REGISTRATION NO. _____

(if appropriate)

TELEPHONE

781/879-1285

Docket Number: _____

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INVENTOR(S)		
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☒ Application Data Sheet. See 37 CFR 1.76 ☐ Drawing(s) Number of Sheets _____ ☒ Specification (e.g. description of the invention) Number of Pages <u>6</u>	☐ CD(s). Number of CDs _____ ☐ Other (specify) _____
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Page 2 of 2

PTO/SB/16 (12-08)

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SIGNATURE Joseph M. Centofanti Date AUG 16, 2011

TYPED or PRINTED NAME Joseph M. Centofanti REGISTRATION NO. _____
(if appropriate)

TELEPHONE 781/879-1285 Docket Number: _____

Cobalt Enrichment at Grain Boundaries of Polycrystalline Layered Metal Oxides

David Ofer and Adrian Pullen

TIAX LLC, 35 Hartwell Avenue, Lexington, MA 02421

Abstract:

This invention concerns polycrystalline layered-structure lithiated metal oxides of composition $\text{Li}_{1+x}\text{MO}_{2+y}$ and articles formed therefrom, where $-0.1 \leq X \leq 0.3$, $-0.3 \leq Y \leq 0.3$, M is about 25-97 % Ni, 3-50 % Co, 0-60 % Mn, and 0-10 % additional elements which may comprise one or more of Al, Mg, Ca, Sr, Ba, Zn, Ti, Zr, Cr, Mo, Fe, V, Si, Ga and B, said polycrystalline layered-structure lithiated metal oxides having enrichment of Co in the grain boundary regions between primary crystallites, i.e., concentration of Co is higher in the grain boundary regions than in the bulk of the primary crystallite.

These compositions with Co-enriched grain boundaries between primary crystallites exhibit dramatically enhanced electrochemical cycling stability as lithium is de-intercalated from and re-intercalated into the crystal lattice.

Background/Prior Art

Layered structure lithium nickelate (LiNiO_2)-based materials have been sought after for Lithium-ion battery cathodes because they generally have lower cost, higher capacity and higher rate capability than the historically predominant LiCoO_2 cathode material. However, pure LiNiO_2 materials exhibit poor electrochemical stability and cycling performance. It has been found that by substituting varying amounts of other metals for some or much of the Ni in M of LiMO_2 -based compositions, some of the capacity and cost benefits of LiNiO_2 materials can be obtained with much improved electrochemical cycling stability, and accordingly a wide variety of Ni-containing layered-structure $\text{Li}_{1+x}\text{MO}_{2+y}$ materials with various compositions of metals M have been developed. In particular, Co incorporation is found to enhance electrochemical cycling stability, and is readily achieved because of the favorable solid-state solution interaction of LiCoO_2 with LiNiO_2 . However, Co is generally considerably more costly than Ni or Mn, the other principal transition metals used in formulating LiMO_2 -based layered structure materials, and Co incorporation in LiNiO_2 generally results in some diminution of the latter material's high capacity and rate capability. Therefore it is desirable to use only as much Co as is required to confer the needed stability to a Ni-based LiMO_2 material.

Ni-based layered materials of the LiMO_2 type are dense polycrystalline agglomerates of submicron-sized primary crystallites. It has been shown that in polycrystalline Ni-containing LiMO_2 -based materials having 2D $\alpha\text{-NaFeO}_2$ -type layered structure, electrochemical cycling induces the 2D layers of transition metal atoms at the crystallite grain boundaries (edges) to reorganize into a NiO-type rock salt structure and/or to be reduced in oxidation state. These transformations are also associated with the appearance of cracks between adjacent crystallites. The resulting NiO-type phases and cracks in

grain boundaries between crystallites can be expected to significantly degrade the electrochemical performance of these materials. This work has also shown that whereas Ni atoms at the grain boundaries undergo reduction in oxidation state as part of these transformations, Co atoms in the grain boundary regions do not, suggesting that it is primarily the Ni atoms that are mobile and that drive the structural rearrangements in the grain boundary regions.

The above observations indicate that Co substitution enhances electrochemical cycling by stabilizing the grain boundaries of polycrystalline Ni-based layered LiMO_2 . Therefore Co-substituted Ni-based LiMO_2 materials with Co preferentially concentrated in the intercrystallite grain boundary regions can obtain enhanced cycling stability while having lower Co levels in the bulk crystallites and minimal overall cobalt content compared to Ni-base LiMO_2 materials with uniformly distributed Co. Materials with such Co-enriched grain boundaries obtain the stabilizing benefits of Co substitution while minimizing the detrimental impacts of Co substitution on capacity, rate capability and cost.

Co-enriched grain boundary materials are explicitly distinguished from “core/shell” materials in which Ni-based LiMO_2 secondary particles are coated with a Co-based material. In these structures, only the secondary particle is coated with Co-based LiMO_2 , ostensibly to reduce the interaction between Ni and electrolyte. Such core/shell materials do not have stabilizing intercrystallite Co enrichment within the bulk of the secondary particle.

TIAX Invention

The object of this invention is to provide for enhanced discharge capacity and electrochemical cycling stability in lithium-ion batteries of a polycrystalline Ni-containing LiMO_2 -based material having 2D $\alpha\text{-NaFeO}_2$ -type layered structure, where M represents multiple elements. This objective is achieved by enriching the grain boundary regions of such polycrystalline materials with Co. Selective enrichment of the grain boundaries with Co minimizes the overall amount of Co that must be incorporated in such materials in order to enhance their electrochemical cycling. The benefits of minimizing a material's overall Co content are reduced cost together with enhanced capacity and rate capability. At the same time, such Co-enrich grain boundary materials have more Co concentrated in the grain boundaries, with attendant benefits to cycling stability, than do many materials having much higher overall Co content uniformly distributed in the bulk.

The polycrystalline material of this present invention can have the composition $\text{Li}_x\text{Q}_y\text{MO}_2$, wherein,

x is in the range $-0.1 \leq x \leq 1.2$

y is in the range $-0.00 \leq y \leq 0.2$

Q represents one or more elements selected from the list: Mg, Sr, Ca, Cu, Zn, etc., and is predominantly associated with the lithium-site of the crystal structure

M represents one or more elements selected from the list: Ni, Co, Mn, Al, Mg, Ca, Sr, Ba, Zn, Ti, Zr, Cr, Mo, Fe, V, Si, Ga and B. M can comprise 25-97 % Ni, 3-50 % Co, 0-60 % Mn, and 0-10 % additional elements selected from the list Al, Mg, Ca, Sr, Ba, Zn, Ti, Zr, Cr, Mo, Fe, V, Si, Ga and B.

The inter-crystallite region of this polycrystalline material has a higher concentration of Cobalt than the bulk.

Example 1

A sample of polycrystalline 2D α -NaFeO₂-type layered structure having overall composition $\text{Li}_{0.932}\text{Mg}_{0.023}\text{Ni}_{0.905}\text{Co}_{0.072}\text{O}_{1.955}$ with Co-enriched grain boundaries was prepared, and thinly sectioned (100-150 nm thick) particles of the material were analyzed by scanning transmission electron microscopy (STEM) with energy dispersive x-ray analysis (EDX). Figure 1 shows a low resolution image of an approximately 11 micron diameter secondary particle, and shows the secondary particle to be composed of densely agglomerated primary particles or crystallites.

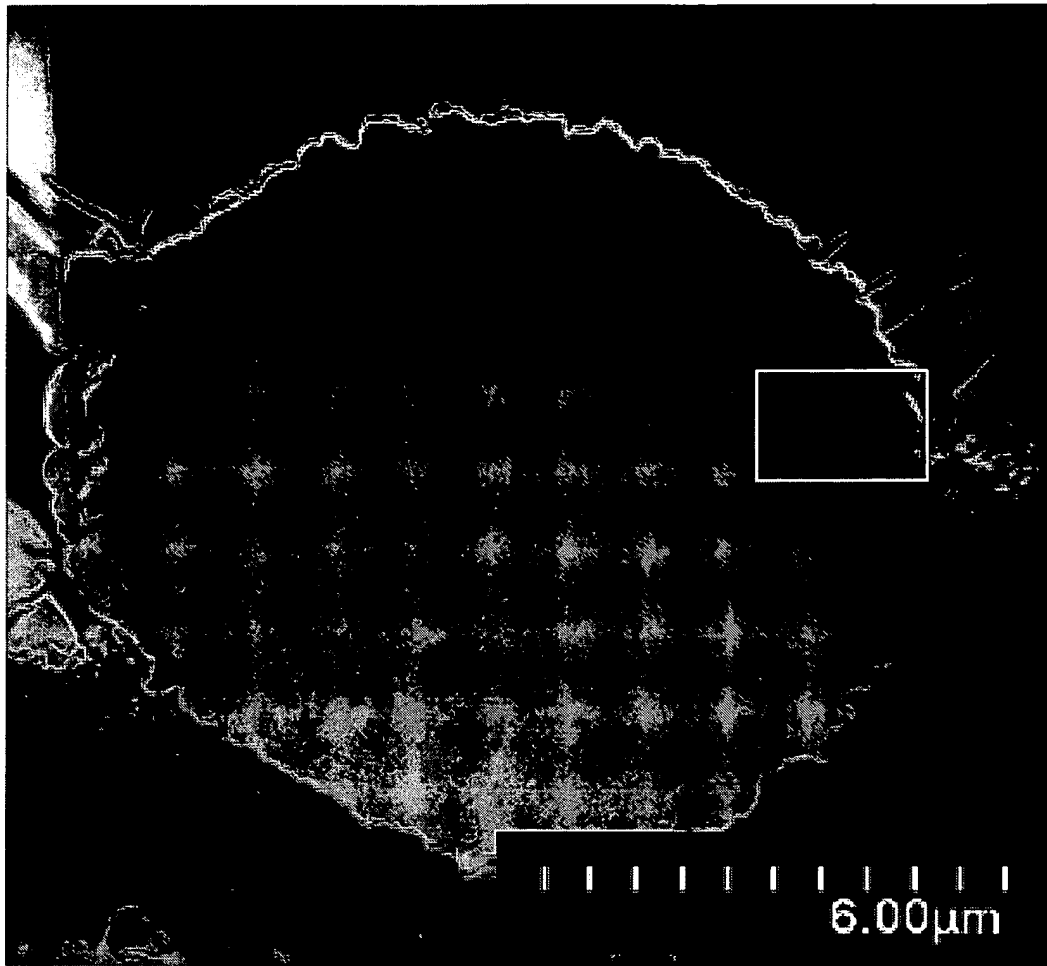


Figure 1. Cross section of $\text{Li}_{0.932}\text{Mg}_{0.023}\text{Ni}_{0.905}\text{Co}_{0.072}\text{O}_{1.955}$ particle.

The white box in Figure 1 shows an area of the cross-sectioned particle that was analyzed by high resolution STEM with EDX. Figure 2 shows the higher magnification STEM image of the selected area, together with the overall EDX spectrum for the imaged area.

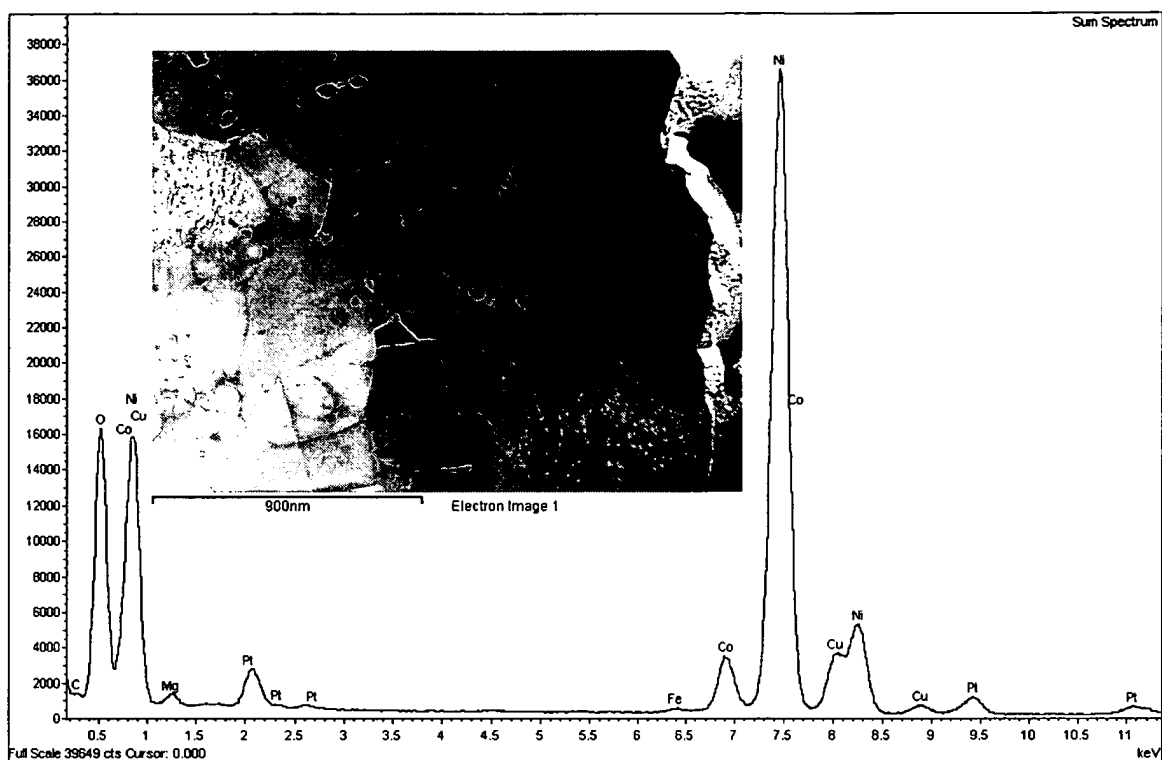


Figure 2. STEM image (inset photograph)_and overall EDX analysis of selected area in $\text{Li}_{0.932}\text{Mg}_{0.023}\text{Ni}_{0.905}\text{Co}_{0.072}\text{O}_{1.955}$ particle cross-section.

The image in Figure 2 further reveals the polycrystalline nature of the material. Integration of the EDX spectrum gives Co/Ni ratio of 0.079, in agreement with the overall material composition. However, high resolution EDX mapping of the same area, shown in Figure 3, reveals that, whereas Ni is uniformly distributed within the crystallites, Co is concentrated at the grain boundaries of the crystallites.

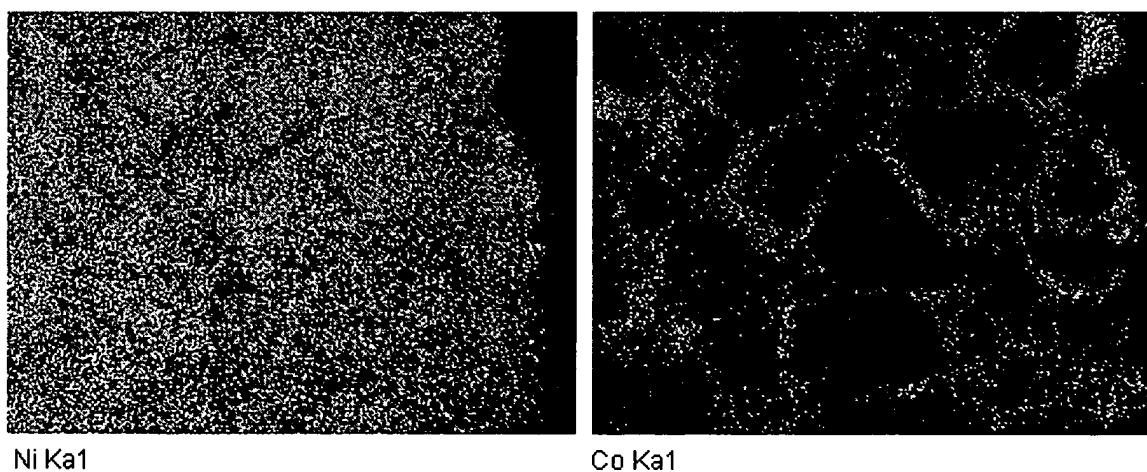


Figure 3. High resolution EDX mapping of Ni (left) and Co (right) for the cross-section STEM image shown in Figure 2.

Example 2

Two samples of polycrystalline 2D α -NaFeO₂-type layered structure having overall composition $\text{Li}_{(0.899)}\text{Mg}_{(0.022)}\text{Ni}_{(0.87)}\text{Co}_{(0.105)}\text{O}_{(1.89)}$ were made, one with Co-enriched grain boundaries and one with a uniform distribution of Cobalt. The materials were blended with PVDF binder and conductive carbon in a slurry of NMP solvent and coated onto an aluminum foil current collector. Cathode electrodes were then punched out of the foil and combined with lithium foil anodes, glass fiber separators and carbonate based electrolytes in a coin cell format for electrochemical testing. The cells were cycled through a series of charge and discharge cycles at a range of current rates and the total electrochemical discharge capacity at each rate recorded. The results of these tests are shown in the table below. The material with grain boundary Co enrichment has higher overall capacity at all rates than the uniform Co distribution material, and also has higher rate capability as indicated by Ratio of 5C to C/5.

Table 1: Half Cell results for two types of 12% Co in LiNiO₂ type of material

Cathode Type	Discharge Capacity (mAh/g)			Ratio 5C to C/5
	C/20	C/5	5C	
Co Enriched grain boundaries	214	201	168	84%
Uniform Co distribution	207	196	144	73%

Claims:

1. A polycrystalline layered metal oxide with composition $\text{Li}_{1+x}\text{MO}_{2+y}$, where - $0.1 \leq X \leq 0.3$, $-0.3 \leq Y \leq 0.3$, and M stands for one or more elements. M can comprise 25-97 % Ni, 3-50 % Co, 0-60 % Mn, and 0-10 % additional elements selected from the list Al, Mg, Ca, Sr, Ba, Zn, Ti, Zr, Cr, Mo, Fe, V, Si, Ga and B. These additional elements can reside either in the transition metal layer or in the lithium layer of the polycrystalline material with the α -NaFeO₂-type layered structure.
2. The polycrystalline material of Claim 1, having Cobalt enrichment in the regions between two crystals, that is Cobalt enrichment in the grain boundary regions. Cobalt enrichment at the primary particle grain boundaries can be measured by a high resolution analytical technique.



"grain boundary" provisional

Nadine Andon to: Elaine McLaughlin
Cc: Joe Centofanti

08/15/2011 11:28 AM

Elaine,

Please make the following minor edits to the attached document:

- ✓ 1. Page One: remove lines 1, 2, 3 and 4 (i.e., "confidential" ; the May date; "not to be disclosed,..."). The first line on the first page should be the title of the invention.
2. All pages: remove lines 1 and 2 (i.e., "confidential..." "not to be disclosed...")
- ✓ 3. Last page: remove the word, "draft", before the word, "claims".

Please prepare the necessary paperwork to file this document as a provisional application for Joe's signature. The inventors are:

1. David Ofer of Needham, MA
2. Adrian Pullen of Jamaica Plain, MA

The invention was **not** reduced to practice in any Government contract.

Please let me know the date this application is filed.

Thanks,
Nadine



Cobalt Enrichment Provisional Aug10_2010.doc

PATENT APPLICATION SERIAL NO. _____

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PATENT AND TRADEMARK OFFICE
FEE RECORD SHEET

08/18/2011 SZEWDIE1 00000034 121675 61575115
01 FC:2005 110.00 DA

PTO-1556
(5/87)