(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
30 October 2014 (30.10.2014)

WIPO | PCT

(10) International Publication Number
WO 2014/174527 A1

(51) International Patent Classification:
C22B 7/04 (2006.01) *C01F 7/44* (2006.01)
B82Y 30/00 (2011.01)

(21) International Application Number:
PCT/IN2014/000197

(22) International Filing Date:
28 March 2014 (28.03.2014)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
1469/MUM/2013 22 April 2013 (22.04.2013) IN

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(81) Designated States *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IR, IS, JP, KE, KG, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States *(unless otherwise indicated, for every kind of regional protection available)*: ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

Published:

— *with international search report (Art. 21(3))*

[Continued on next page]

(54) Title: METHOD FOR RECOVERY OF ALUMINA FROM ALUMINIUM DROSS

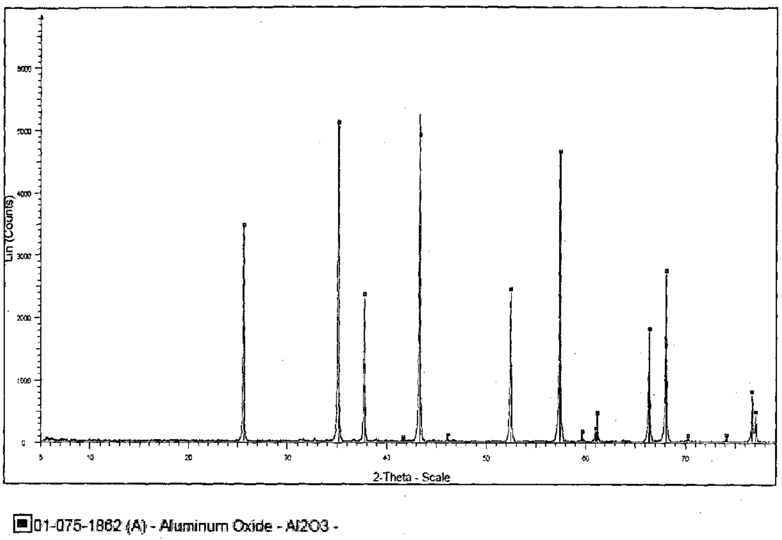

Figure 2

(57) **Abstract**: A method for recovery of a-alumina from aluminium dross is disclosed. The method comprises of grinding aluminium dross to obtain particles having average particle size in the range of 80-100 micron, leaching aluminium dross with an acid to obtain a leach liquor having precipitated silica, separating the silica from the leach liquor to obtain a liquid phase, treating the liquid phase with an aqueous ammonia at acidic pH to facilitate precipitation of iron hydroxide, separating the iron hydroxide from the liquid phase to obtain an iron free liquid phase, treating the iron free liquid phase with aqueous ammonia at alkaline pH to facilitate precipitation of aluminium hydroxide; separating and calcining the aluminium hydroxide to obtain α-alumina having size in the range of 200-700 nm.

WO 2014/174527 A1

— *before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments (Rule 48.2(h))*

METHOD FOR RECOVERY OF ALUMINA FROM ALUMINIUM DROSS

The present disclosure provides a method for recovery of α-alumina from aluminium dross. Particularly, a method of recovery of nano alumina from aluminium dross is disclosed.

Background

Aluminium dross is residue from primary and secondary aluminium production and is generated during aluminium smelting process. Aluminium dross is classified into white and black dross, according to its metal content. White dross is of higher metal content and it is produced from primary and secondary aluminium smelters. White dross may contain 15% to 70% recoverable metallic aluminium. Black dross has a lower metal content and is generated during aluminium recycling. Black dross contains recoverable aluminium in a range of 2% to 15%. Black dross is mainly mixture of aluminium oxide and other compounds such as AlN, Al_4C_3, MgF_2, $NaAlCl_4$, $KNaCl_4$, SiO_2, Fe_2O_3 and MgO etc.

In general, aluminium dross is processed in rotary kilns to recover the aluminium and the resultant salt cake is sent to landfills as waste. Such disposal is increasingly facing environmental problems since salts can leach from the aluminium dross and pass into the soil below. In addition to this when aluminium dross comes in contact with water it emits harmful gases such as NH_3, CH_4, PH_3, H_2, H_2S, etc. It is known in art that the dross produced by the aluminium industry, although a waste, contain significant amount of valuable element such as aluminium oxide, which can be recovered and reused.

A process for production of G-alumina from waste aluminium dross is reported in Das *et al*; "Production of G-alumina from Waste Aluminium Dross", Minerals

Engineering 20 (2007) 252–258.This document discloses a process for obtaining G-alumina from aluminium dross and the percent recovery of G-alumina is less than 90%. Furthermore, the G-alumina obtained by the process disclosed in said document does not have nanoscale dimensions.

US7651676 describes a process for preparing aluminium chloride by leaching aluminium dross residues with hydrochloric acid or sulphuric acid to obtain a mixture comprising a solid and a liquid. Further,the liquid obtained is hydrochlorinated to precipitate aluminium chloride. The process further comprises of converting the aluminium chloride into alumina.

The method known in the art either use an additional calcining step or other multiple steps to obtain alumina. There is therefore a need for a simpler and economical process for recovery of alumina from aluminium dross.

Summary

A method for recovery of α-alumina from aluminium dross is disclosed. The method comprises of grinding aluminium dross to obtain particles having average particle size in the range of 80-100 micron, leaching aluminium dross with an acid to obtain a leach liquor having precipitated silica, separating the silica from the leach liquor to obtain a liquid phase, treating the liquid phase with an aqueous ammonia at acidic pH to facilitate precipitation of iron hydroxide, separating the iron hydroxide from the liquid phase to obtain an iron free liquid phase, treating the iron free liquid phase with aqueous ammonia at alkaline pH to facilitate precipitation of aluminium hydroxide; separating and calcining the aluminium hydroxide to obtain α-alumina having size in the range of 200-700 nm.

Brief Description of Drawings

Figure 1: illustrates scanning electron micrograph of α-alumina obtained in accordance with the disclosed method.

Figure 2: illustrates phase analysis using powder X-ray diffraction of α-alumina obtained by the disclosed method.

Detailed Description

For the purpose of promoting an understanding of the principles of the invention, reference will now be made to embodiments and specific language will be used to describe the same. It will nevertheless be understood that no limitation of the scope of the invention is thereby intended, such alterations and further modifications in the disclosed process, and such further applications of the principles of the invention therein being contemplated as would normally occur to one skilled in the art to which the invention relates.

It will be understood by those skilled in the art that the foregoing general description and the following detailed description are exemplary and explanatory of the invention and are not intended to be restrictive thereof.

Reference throughout this specification to “one embodiment” “an embodiment” or similar language means that a particular feature, structure, or characteristic described in connection with the embodiment is included in at least one embodiment of the present invention. Thus, appearances of the phrase “in one embodiment”, “in an embodiment” and similar language throughout this specification may, but do not necessarily, all refer to the same embodiment.

The term "nano alumina" as referred to herein means alumina particles having dimensions of nanoscale (1-1000nm).

The present disclosure provides a method for recovery of α-alumina from aluminium dross. Particularly, a method of recovery of nano alumina from aluminium dross is disclosed. Nano alumina has better properties including reactivity as compared to α-alumina not having dimensions of nanoscale. The method comprises of grinding of aluminium dross to obtain an average particle size in a range of 80-100 micron. The ground aluminium dross is subjected to leaching at high temperature with an acid to obtain leach liquor having aluminium and iron in a dissolved state and a precipitate of silica. The silica thus obtained is separated from the leach liquor. The liquid phase obtained after separation of silica is treated with aqueous ammonia at an acidic pH to facilitate precipitation of iron as iron hydroxide [$Fe(OH)_3$]. The iron hydroxide is separated to obtain iron free liquid phase. Thus obtained iron free liquid phase is treated with aqueous ammonia at an alkaline pH to facilitate precipitation of aluminium hydroxide. The method further comprises of separating and calcining the aluminium hydroxide to obtain α-alumina having size in a range of 200 nm to 700 nm.

In accordance with an embodiment, separation of silica from the leach liquor is carried out by filtration.

In accordance with an embodiment, the acidic pH at which the liquid phase is treated with aqueous ammonia is in a range of 4-6.

In accordance with an embodiment, separation of iron hydroxide from the liquid phase is carried out by filtration.

In accordance with an embodiment, the alkaline pH at which the iron free liquid phase is treated with aqueous ammonia is in a range of 7.5-8.5.

In accordance with an embodiment, separation of aluminium hydroxide is carried out by any known methods such as filtration. The separated aluminium hydroxide is subjected to washing and drying. Thus obtained dried aluminium hydroxide is calcined at a temperature in a range of 1100°C to 1200°C to obtain α-alumina.

In accordance with an embodiment, the aluminium dross may be a black dross comprising Al_2O_3, AlN, SiO_2 and Fe_2O_3. In accordance with an embodiment, the black dross comprises of Al_2O_3 in a range of 60-80%, AlN in a range of 15-30%, SiO_2in a range of 0-10% and Fe_2O_3in a range of 0-5%. By way of specific example, the black dross comprises of 70% of Al_2O_3, 25% of AlN, 3.5% of SiO_2 and 1.5% of Fe_2O_3.

In accordance with an embodiment, the grinding of aluminium dross is carried out to obtain particle size in a range of 7-1540 micron, with average particle size of 80-100 micron, preferably 90 micron.

In accordance with an embodiment, acid utilized for leaching of aluminium dross is sulphuric acid having concentration in a range of 30%-50% (v/v).

In accordance with an aspect, leaching of aluminium dross with sulphuric acid is carried out at a high temperature in a range of 170 °C -230 °C for a time period in a range of 10- 15 hours. Leaching of aluminium dross at such a temperature for prolonged duration of time (i.e. 10-15 hrs) facilitates production of highly pure α-alumina with high percentage yield.

In accordance with an embodiment, aqueous ammonia used for precipitation of iron hydroxide and aluminium hydroxide has a concentration in a ratio of 1:1.

In accordance with an embodiment, drying of aluminium hydroxide precipitate prior to calcination is carried out at a temperature in a range of 100 °C to 200 °C.

In accordance with an embodiment, calcination of dried aluminium hydroxide precipitate is carried out for a time period in a range of 15 minutes to 1 hour to obtain α-alumina having size in the range of 200 nm to 700 nm.

In accordance with an embodiment, percentage yield of α-alumina by the method disclosed herein is >90.

In accordance with an embodiment, the α-alumina obtained by the method disclosed herein is highly crystalline single phase α-alumina (as illustrated in Figure 2) having purity of 99.5%.

The following example(s) of method for recovery of α-alumina from aluminium dross and/or analysis of alumina obtained from said method are exemplary and should not be understood to be in any way limiting.

Example 1:

The raw aluminium dross used in this study contained 80% alumina, (quantitative XRD study using Rietveld analysis (70% Al2O3 and 20% AlN, i.e 10% Al2O3). 10 gms batch of the aluminium dross was taken and subjected to leaching, precipitation followed by calcination at 1200°C. White powder obtained after calcination was subjected to powder X-ray diffraction which revealed the single phase nature of the material (presence of highly crystalline alumina). The weight of the powder was found to be 5.6 gms indicating 92% alumina extraction efficiency (yield).

Whiteness of the powder sample was found to be 92 when measured using reflectance spectrophotometer.

Example 2:

Purity of synthesised alumina was evaluated using EDTA back titration. For the titration, the sample was prepared by dissolving alumina obtained from the method disclosed in a mixture of acids. In conical flask sample solution was pipetted out along with buffer solution (pH=10) and EDTA (0.01M). Subsequently, the solution was boiled on a hot plate for 5 mins to speed up the formation of Al-EDTA complex. 3-4 drops of Erichrome black T indicator was added and mixed so that the solution appears pure blue in colour. Excess of EDTA was titrated with ZnSO4 solution (0.01M) until the colour changed to purple blue at its endpoint. The purity of alumina was calculated and found to be 99.5%.

Specific Embodiments are Described Below

A method for recovery of α-alumina from aluminium dross comprising grinding aluminium dross to obtain particles having average particle size in the range of 80-100 micron, leaching aluminium dross with an acid to obtain a leach liquor having precipitated silica, separating the silica from the leach liquor to obtain a liquid phase, treating the liquid phase with an aqueous ammonia at acidic pH to facilitate precipitation of iron hydroxide, separating the iron hydroxide from the liquid phase to obtain an iron free liquid phase, treating the iron free liquid phase with aqueous ammonia at alkaline pH to facilitate precipitation of aluminium hydroxide; and

separating and calcining the aluminium hydroxide to obtain α-alumina having size in the range of 200-700 nm.

Such method(s), wherein leaching of aluminium dross is carried out at a temperature in the range of 170°C – 230°C for a time period in the range of 10-15 hrs.

Such method(s), wherein the acid is suphuric acid having concentration in the range of 30%-50% (v/v).

Such method(s), wherein the acidic pH at which the liquid phase is treated with aqueous ammonia is in the range of 4-6.

Such method(s), wherein the alkaline pH at which the iron free liquid phase is treated with aqueous ammonia is in the range of 7.5 to 8.5.

Such method(s), wherein calcining is carried out at a temperature in the range of 1100°C – 1200°C.

Industrial Application

The method for recovery of α-alumina from aluminium dross described above is easy to perform, economical and utilizes a waste generated during aluminium smelting process as a starting material. The α-alumina obtained from the method described above is highly crystalline single phase α-alumina having percentage purity of 99.5. Further, α-alumina obtained has a size in the range of 200 nm to 700 nm. Nano alumina even if used in small amounts significantly improves properties of a desired product.

We Claim:

1. A method for recovery of α-alumina from aluminium dross comprising:

   grinding aluminium dross to obtain particles having average particle size in the range of 80-100 micron;

   leaching aluminium dross with an acid to obtain a leach liquor having precipitated silica;

   separating the silica from the leach liquor to obtain a liquid phase;

   treating the liquid phase with an aqueous ammonia at acidic pH to facilitate precipitation of iron hydroxide;

   separating the iron hydroxide from the liquid phase to obtain an iron free liquid phase;

   treating the iron free liquid phase with aqueous ammonia at alkaline pH to facilitate precipitation of aluminium hydroxide; and

   separating and calcining the aluminium hydroxide to obtain α-alumina having size in the range of 200-700 nm.

2. A method for recovery of α-alumina from aluminium dross as claimed in claim 1, wherein leaching of aluminium dross is carried out at a temperature in the range of 170°C – 230°C for a time period in the range of 10-15 hrs.

3. A method for recovery of α-alumina from aluminium dross as claimed in claim 1, wherein the acid is suphuric acid having concentration in the range of 30%-50% (v/v).

4. A method for recovery of α-alumina from aluminium dross as claimed in claim 1, wherein the acidic pH at which the liquid phase is treated with aqueous ammonia is in the range of 4-6.

5. A method for recovery of α-alumina from aluminium dross as claimed in claim 1, wherein the alkaline pH at which the iron free liquid phase is treated with aqueous ammonia is in the range of 7.5 to 8.5.

6. A method for recovery of α-alumina from aluminium dross as claimed in claim 1, wherein calcining is carried out at a temperature in the range of 1100°C – 1200°C.

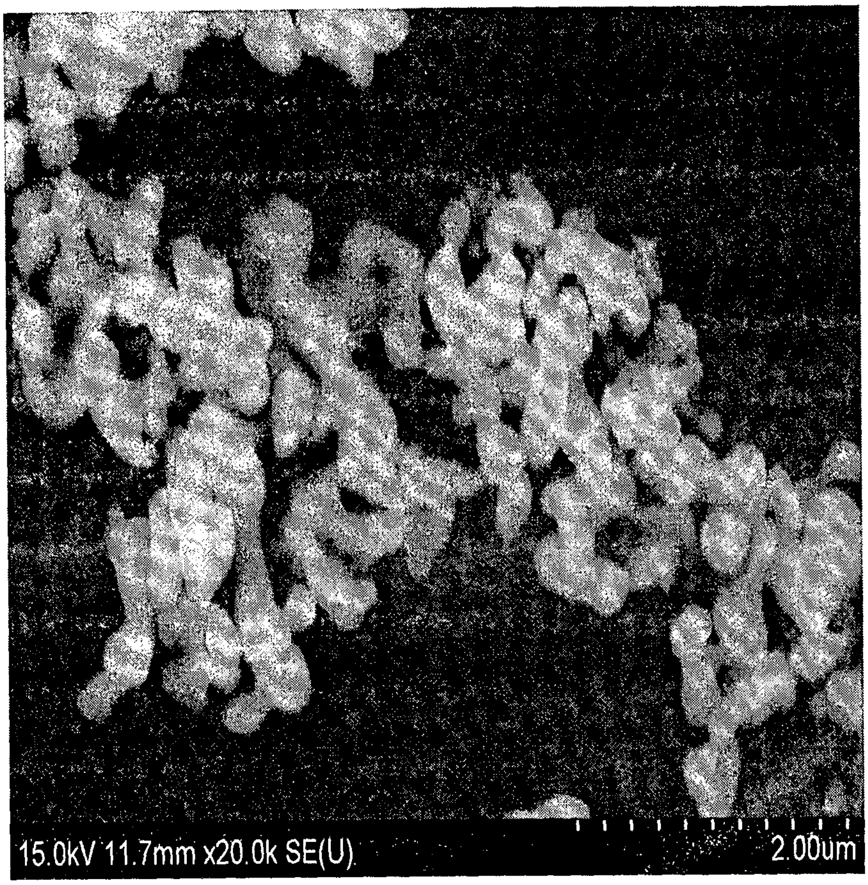

Figure 1

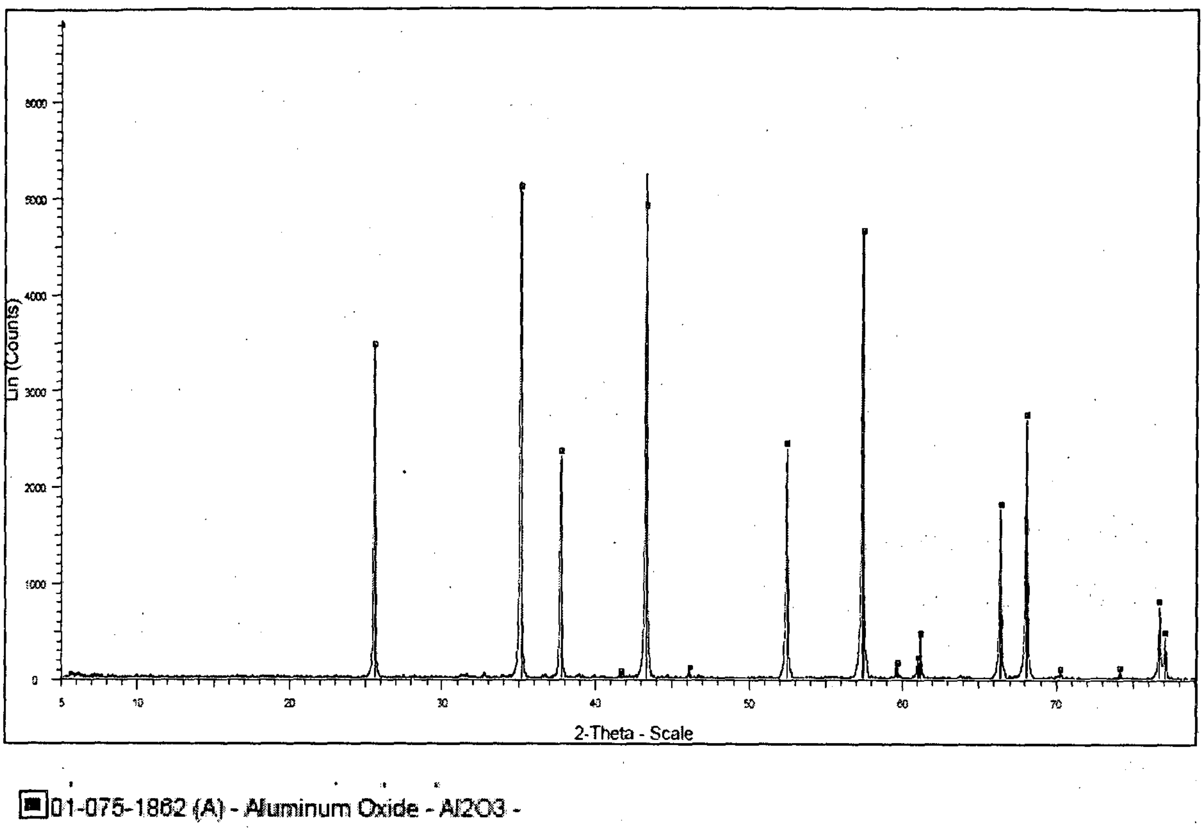
Lin (Counts)
6000
5000
4000
3000
2000
1000
0
5
10
20
30
40
50
60
70
2-Theta - Scale
■ 01-075-1862 (A) - Aluminum Oxide - Al2O3 -

Figure 2

INTERNATIONAL SEARCH REPORT

International application No
PCT/IN2014/000197

A. CLASSIFICATION OF SUBJECT MATTER
INV. C22B7/04 B82Y30/00 C01F7/44
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)
C22B B82Y C01F

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, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DAS ET AL: "Production of eta-alumina from waste aluminium dross", MINERALS ENGINEERING, PERGAMON PRESS , OXFORD, GB, vol. 20, no. 3, 9 February 2007 (2007-02-09), pages 252-258, XP005880787, ISSN: 0892-6875, DOI: 10.1016/J.MINENG.2006.09.002 cited in the application the whole document ----- -/--	1-6

☒ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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Date of the actual completion of the international search 20 August 2014	Date of mailing of the international search report 08/09/2014
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Authorized officer Swiatek, Ryszard

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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DASH B ET AL: "Acid dissolution of alumina from waste aluminium dross", HYDROMETALLURGY, ELSEVIER SCIENTIFIC PUBLISHING CY. AMSTERDAM, NL, vol. 92, no. 1-2, 1 May 2008 (2008-05-01), pages 48-53, XP022603241, ISSN: 0304-386X, DOI: 10.1016/J.HYDROMET.2008.01.006 [retrieved on 2008-01-26] the whole document -----	1-6
A	Liu Qingsheng ET AL: "High Purity Alumina Powders Extracted from Aluminum Dross by the Calcining-Leaching Process" In: "Light Metals 2011", 28 February 2011 (2011-02-28), John Wiley & Sons, Inc., Hoboken, NJ, USA, XP055135313, ISBN: 978-1-11-802935-0 pages 197-200, DOI: 10.1002/9781118061992.ch34, the whole document -----	1-6
A	US 2003/185746 A1 (KAJIHARA KAZUHISA [JP] ET AL) 2 October 2003 (2003-10-02) the whole document -----	1-6

1

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/IN2014/000197

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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