NOTICE OF FILING

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File Title: FORTESCUE LIMITED ACN 002 594 872 & ORS v ELEMENT ZERO PTY

LIMITED ACN 664 342 081 & ORS

Registry: NEW SOUTH WALES REGISTRY - FEDERAL COURT OF AUSTRALIA



Sia Lagos

Registrar

Important Information

This Notice has been inserted as the first page of the document which has been accepted for electronic filing. It is now taken to be part of that document for the purposes of the proceeding in the Court and contains important information for all parties to that proceeding. It must be included in the document served on each of those parties.

The date of the filing of the document is determined pursuant to the Court's Rules.



Form 59 Rule 29.02(1)

Affidavit

No. NSD 527 of 2024

Federal Court of Australia

District Registry: New South Wales

Division: General

FORTESCUE LIMITED (ACN 002 594 872) and others

Applicants

ELEMENT ZERO PTY LIMITED (ACN 664 342 081) and others

Respondents

Affidavit of: Paul Alexander Dewar

Address: Level 4, 7 Macquarie Place, Sydney NSW 2000

Occupation: Lawyer

Date: 18 September 2025

Document number	Details	Paragraph	Page
1	Eleventh Affidavit of Paul Alexander Dewar affirmed on 18 September 2025	1	2
2	Confidential Annexure PAD-76, being copy of the letter from G+T to DCCL dated 9 September 2025.	6	11
3	Annexure PAD-77 , being a copy of an email from DCCL to G+T dated 16 September 2025.	8	14
4	Annexure PAD-78 , being a copy of the duplicate analysis prepared by TransPerfect.	12(b)	16
5	Confidential Annexure PAD-79, being a copy of the 2022 Provisional	13(a)	20
6	Annexure PAD-80, being a copy of the PCT (WO 2024/082020)	13(b)	41
7	Annexure PAD-81, being a copy of the 2023 Provisional (AU 2023902103)	13(c)	75

Filed on behalf of Fortescue Limited and others, the Applicants Prepared by Paul Alexander Dewar, Principal Lawyer

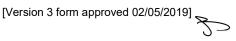
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Document number	Details	Paragraph	Page
8	Confidential Annexure PAD-82, being a copy of BWJ.5000.0001.8908	21	108
9	Confidential Annexure PAD-83, being a bundle of documents discovered by the Third Respondent.	21	112
10	Confidential Annexure PAD-84, being a bundle of documents discovered by the Third Respondent.	26(b)	138
11	Confidential Annexure PAD-85, being a copy of BWJ.5000.0003.4277 and BWJ.5000.0003.4278	27	210

- I, PAUL ALEXANDER DEWAR, of Level 4, 7 Macquarie Place, Sydney, New South Wales, 2000, Lawyer, affirm:
- 1. This is my eleventh affidavit in this proceeding. I adopt the definitions I used in my Tenth Affidavit affirmed on 19 August 2025 (**Dewar 10**).
- 2. I have practised in the field of commercial litigation for 27 years, including over 15 years in the conduct of patent litigation.
- I am authorised to make this affidavit on behalf of Fortescue. I am not instructed, and do
 not intend in this affidavit, to waive privilege on behalf of Fortescue, nor do I have the
 authority to do so.

Corrections to Dewar 10

- 4. Since affirming Dewar 10, I have reviewed paragraphs 67, 155, 197 and 225 of that affidavit, and the table of contents, and wish to make the following corrections:
 - (a) in paragraph 67, the reference in the third line to "does not include" should read "includes";
 - (b) in paragraph 155, second sentence, the references to "paragraphs [259] [265]" should read "paragraphs [261] [267]";
 - (c) in paragraph 197, second and third sentences, the references to "sections J.2 to J.23" should read "sections G.2 to G.22";
 - (d) in paragraph 225, the error reference should be to Category "16"; and
 - (e) document numbers 27, 28 and 33 in the table of contents should not be marked "Confidential".

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Further documents produced by the Respondents

- 5. On 6 August 2025, the Third Respondent produced 13 further documents, in response to notified deficiencies #464 and #468-470 relating to Categories 11(e),(f).
- On Thursday 9 September 2025, G+T wrote to DCCL in relation to further discovery documents to be provided to DCCL. Annexed and marked Confidential PAD-76 is a copy of that letter.
- 7. On Thursday 11 September 2025, the EZ Respondents produced 565 further documents to DCCL.
- 8. On Tuesday 16 September 2025, DCCL wrote to G+T requesting an explanation as to which of the documents were produced on the basis they had been produced by the Third Respondent pursuant to categories 11(d) and 11(f), and for the remainder, to identify which documents were produced in response to a particular notified deficiency. Annexed and marked PAD-77 is a copy of that email.
- 9. At the date of swearing this affidavit, DCCL has not received a response from G+T.
- 10. The Respondents have not served any supplementary lists of discovery documents further to paragraphs 5 and 7 above.
- 11. I caused a team of solicitors at DCCL to commence reviewing the documents produced by the Respondents as referred in paragraphs 5 and 7 above. In the limited time available before the swearing of this affidavit, it has not been possible to manually review those documents.
- 12. In respect of the EZ Respondents' production of 565 documents, I caused the Applicants' e-discovery provider, Transperfect, to conduct a file name comparison of (1) the 565 documents discovered by the EZ Respondents on 11 September 2025, against (2) documents earlier discovered by the Third Respondent in response to the discovery categories, to identify the categories to which the EZ Respondents' further production relate. I am informed by Christopher Lichtenberg at TransPerfect that:
 - (a) approximately half of the 565 documents could not be compared (the analysis could not be run for the email files provided);
 - (b) of the files that could be compared, 142 correspond with documents discovered by the Third Respondent in response to Categories 11(a), 11(b), 11(c), 11(d),

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11(e), 11(f), 12 and 13. Annexed and marked **PAD-78** is a copy of the duplicate analysis prepared by Mr Lichtenberg.

2022 Provisional, PCT, 2023 Provisional

- Annexed to this affidavit and marked:
 - (a) Confidential PAD-79 is a copy of provisional patent application no. AU 2022903090 entitled "METHOD OF ORE PRQCESSING" (2022 Provisional) filed on 20 October 2022, discovered by the Third Respondent (BWJ.5000.0004.1222);
 - (b) **PAD-80** is a copy of PCT patent application no. WO 2024/082020 entitled "METHOD OF ORE PRQCESSING" (**PCT**), which claims priority from the 2022 Provisional, was filed 19 October 2023, and was published 25 April 2024, discovered by the EZ Respondents (EZR.0007.0001 .0002);
 - (c) PAD-81 is a copy of Australian provisional patent application no. AU 2023902103 entitled "ORE PROCESSING METHOD FOR METAL RECOVERY" filed on 30 June 2023 (2023 Provisional), discovered by the EZ Respondents (EZR.0003.0002.0329).
- 14. Deficiency #497-499 relate to Examples 1, 2 and 3 of the 2022 Provisional:
 - (a) I have caused Kevin Huang, lawyer from DCCL, and Rohit Dighe, senior associate from DCCL, to review the Respondents' materials discovered in response to Categories 11(c), (e) and (f) dated before 20 October 2022 (i.e. the filing date of the 2022 Provisional).
 - (b) I am in informed by Mr Huang and Mr Dighe based on their review of the documents, and believe, that there are no documents that record the experiments referred to in Examples 1 to 3 of the 2022 Provisional.
- 15. Deficiency #14 relates to Examples 1 10 of the PCT:
 - (a) I have caused Mr Huang and Mr Dighe to search the Respondents' discovery materials produced in response to Categories 11(c), (e) and (f) dated between

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- 21 October 2022 and 19 October 2023 (i.e. between the filing of the 2022 Provisional, and the PCT).
- (b) I am in informed by Mr Huang and Mr Dighe based on their review of the documents, and believe, that there are no documents that record the experiments referred to in Examples 1 to 3, 6, 8 to 10 of the PCT.
- 16. Deficiency #15 relates to Examples 2 and 3 of the 2023 Provisional:
 - (a) I have caused Mr Huang to search the Respondents' discovery materials produced in response to Categories 11(c), (e) and (f) dated before 30 June 2023 (i.e. before the filing of the 2023 Provisional).
 - (b) I am in informed by Mr Huang based on his review of the documents, and believe, that the only records of Example 2 and 3 are summaries of the experiment, as opposed to a documents recording the experimentation relevant to Examples 2 and 3 of the 2023 Provisional.

Reply to the Dunn Affidavit

- 17. I refer to the seventh affidavit of Rebecca Mary Dunn sworn 9 September 2025 (**Dunn 7**), and the eighth affidavit of Ms Dunn sworn 16 September (**Dunn 8**). Where I do not comment on an aspect of Dunn 7 and Dunn 8 below, that does not mean that I agree with that aspect.
- 18. At paragraphs 33–34 of Dunn 7, Ms Dunn suggests Fortescue had "pre-determined" to allege deficiencies before it had reviewed the EZ Respondents' discovery. That is not correct. The EZ Respondents served their Original Lists of Documents on Fortescue at 3.58 pm on 29 April 2025, and I caused solicitors at DCCL to review those Lists of Documents and it was apparent on their face that:
 - (a) the EZ Respondents had failed to describe in Part 2 of each of their Original Lists of Documents each document in respect of which any EZ Respondent made a claim for privilege (as required by rule 20.17(2)(c) of the FCR);
 - (b) the EZ Respondents had failed to describe in Part 3 of each of their Original Lists of Documents each document that had been but was no longer in each EZ Respondent's control (as required by rule 20.17(2)(b) of the FCR);
 - (c) the EZ Respondents had discovered no documents in answer to Categories 2A(a), 2A(c), 5, 6A, 7, 8, 11(e) and 14 (general deficiency #4); and

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- (d) the EZ Respondents had discovered only one document in answer to Category6 and none of the documents particularised at FASOC [19] (general deficiency #10).
- 19. At paragraphs 47, 52, 55, 56, 58, 60, 61 and 65 of Dunn 7, Ms Dunn states that the EZ Respondents conducted searches over the Seized Materials in relation to Categories 1, 2A(e), 2A(f), 2A(g), 3, 6, 6A and 11(d), respectively. However, Dunn 7 does not disclose that the EZ Respondents conducted searches over the Seized Materials for documents responsive to Categories 2A(a), 2A(b), 2A(c), 2A(d), 5, 7, 8, 11(a), 11(b), 11(c), 11(e), 11(f), 12, 13, 14 and I infer they did not do so.
- 20. At paragraph 47 of Dunn 7, Ms Dunn explains that "[o]nce processed, there were approximately 3 million records in the Seized Materials on which searches described below were run". At paragraph 9(d) of Dunn 8, Ms Dunn states the Seized Materials "contained approximately 7 million individual files". Ms Dunn has not explained the basis for this difference; however it may mean that a material number of records (potentially up to four million records) in the Seized Materials were not searched in order to comply with the discovery categories.
- 21. At paragraph 52 of Dunn 7, Ms Dunn sets out the keywords used to retrieve documents responsive to Category 1. The use of quotation marks indicates to me that the EZ Respondents performed exact phrase searches; that is, the searches do not contain 'wildcard' searches, or otherwise accommodate permutations of a given exact phrase. The Respondents also did not use other reasonable search terms (e.g. "NaOH", "KOH" which are terms for the ionic liquid compositions relevant to these proceedings) or abbreviations (for example, "ILs" for "ionic liquids", as used in email BWJ.5000.0001.8908 by Dr Winther-Jensen, sent to Dr Kolodziejczyk on 20 January 2021, annexed and marked Confidential Annexure PAD-82). This indicates to me a reason to doubt the adequacy of the Respondents' discovery in respect of Category 1. To demonstrate this, I have reviewed 8 documents discovered by the Third Respondent in response to Category 1, a bundle of which is annexed and marked Confidential Annexure PAD-83. I consider each of them to be responsive to Category 1. None contains the search terms mentioned at paragraph 52 of the Dunn Affidavit.
- 22. Discovery category 2A consists of a chapeau description (namely, "All documents, and all documents recording information, copied, taken or otherwise obtained by the Second Respondent or the Third Respondent from Fortescue (including Fortescue's network, systems or devices) in the period from September 2021 to November 2021, including:")

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followed by specific, non-exhaustive examples. Paragraphs 53 to 57 of Dunn 7 refer to searches conducted only with respect to the non-exhaustive examples of **Category 2A**. Ms Dunn does not state that any searches were conducted for documents captured by the chapeau description of category 2A. It is apparent no searches were in fact conducted for documents responsive to the chapeau description of Category 2A having regard to the EZ Respondents' Original and Supplementary Lists of Discovered Documents (see e.g. PAD-61, PAD-62 to Dewar 10). This indicates to me a reason to doubt the adequacy of the Respondents' discovery in respect of Category 2A, because the EZ Respondents have not searched for documents responsive to the chapeau.

- 23. At paragraphs 53 and 106, Ms Dunn states that it was Dr Kolodziejczyk who searched his Gmail account for documents, and determined their relevance, under **Categories 2A(a)-(b)**. Ms Dunn does not engage with the clear misunderstanding of that category by Dr Kolodziejczyk (that the category calls for documents "taken by him to finish off" his work for Fortescue, not the finished work itself) as notified by deficiency #6. For these reasons, I doubt the adequacy of the EZ Respondents' discovery in respect of Categories 2A(a)-(b).
- 24. At paragraph 57 and 60 of Dunn 7, Ms Dunn explains that the EZ Respondents conducted searches responsive to **Category 2A(g)** "by reference the documents particularised in paragraph 20 of the [FASOC]" (paragraph 57), and "for the 106 documents identified by Fortescue as 'First Specified Documents'" (paragraph 60). The effect of this is that the EZ Respondents treated Category 2A(g) as effectively the same as Category 6.
- 25. However, that is not what the Category 2A(g) called for, and categories 2A(g) and 6 are not the same. Category 2A(g), required discovery of: "All documents, and all documents recording information, copied, taken or otherwise obtained by the Second Respondent or the Third Respondent from Fortescue (including Fortescue's network, systems or devices) in the period from September 2021 to November 2021, including....documents by the Third Respondent from his Fortescue email address "bjorn.wintherjensen@fmgl.com.au" to his personal email address *"bjornwj@gmail.com"*" (emphasis added). Dunn 7 does not disclose that the EZ Respondents conducted any searches by reference to the email addresses specified in Category 2A(g) (that is "bjorn.wintherjensen@fmgl.com.au" and "bjornwj@gmail.com"). The omission of a separate search for category 2A(g), and the omission of search terms

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"bjorn.wintherjensen@fmgl.com.au" and "bjornwj@gmail.com", indicate to me reasons to doubt the adequacy of the Respondents' discovery in respect of **Category 2A(g)**.

- 26. At paragraph 61, Ms Dunn states that the EZ Respondents searched for **Category 6A** by key word search for "Fortescue' and 'plant". I consider that search parameter to be inadequate. To demonstrate this, I have caused the search terms "Fortescue" and "plant" to be run across the six documents discovered by the Third Respondent in response to Category 6A (which I too have reviewed and consider to be responsive to the category in that the documents "concern[] the design, engineering, construction, operation and/or feasibility of a green iron pilot plant…from Fortescue"). Of those:
 - (a) only one document contains both "Fortescue" and "plant" (BWJ.5000.0002.8478).
 - (b) the remaining documents would not have responded to Ms Dunn's search. Two documents mention neither "Fortescue" nor "plant" (BWJ.5000.0002.2935 and BWJ.5000.0002.3547); two documents mention only "plant" (BWJ.5000.0002.2983 and BWJ.5000.0002.3379); and a final document mentions "plant" and, although the word "Fortescue" appears, it is not text searchable in that document (BWJ.5000.0002.6637). Copies of those documents are annexed and marked Confidential Annexure PAD-84.

The use of this inadequate search parameter indicates to me a reason to doubt the adequacy of the Respondents' discovery in respect of Category 6A, because responsive documents are likely not to have been retrieved for review.

- 27. At paragraph 66, Ms Dunn states her view that any documents discoverable under Category 11(e) would already be produced under Category 11(f). It is apparent that the EZ Respondents did not conduct a separate search for Category 11(e) documents on that basis. However, Categories 11(e) and 11(f) are not the same. Annexed and marked Confidential Annexure PAD-85 is a copy of documents BWJ.5000.0003.4277 and BWJ.5000.0003.4278, being a copy of the 'Technology Base Presentation' and covering email sent by Dr Winther-Jenssen to Dr Kolodziejcyzyk on 21 May 2022. These are copies of the documents referred to in Dewar 10 paragraph 63(b). They are documents responsive to category 11(e), but not 11(f).
- 28. At paragraphs 82-85 and 87 of Dunn 7, Ms Dunn refers to the 'Technology Base Presentations' and states her view that the Technology Base Presentations are not documents relevant to category 11(f). Ms Dunn did not separately consider that these

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documents were discoverable under Category 11(e). The failure to conduct a separate search for Category 11(e) documents, to which there are responsive documents like **Confidential Annexure PAD-85**, indicates to me a reason to doubt the adequacy of the Respondents' discovery in respect of Category 11(e).

- 29. At paragraph 68, Ms Dunn states that the EZ Respondents discovered documents "applications and patents in which <u>Dr Kolodziejczyk was named as an inventor</u>" under **Category 13**. However, that is not all what the category called for. Category 13 extends to "Copies of all patents and patent applications (or divisional or related patents and patent applications) <u>filed by any of the Respondents</u>..." including "drafts thereof". For example, the "979 Application" identified in discovery category 13(c) is a patent application filed by Element Zero, even though Dr Kolodziejczyk is not named as an inventor.
- 30. Copies and drafts of this application have not been discovered by the EZ Respondents under category 13, but they ought to have been. I have caused a solicitor in the employ of DCCL to search for "divisional or related patents and patent applications" of the 979 Application. I am informed by Kevin Huang, and believe, that there is a PCT Application no. WO2025118033 related to the 979 Application, but copies and drafts of it have not been discovered. Additionally, Ms Dunn states at paragraph 68 that responsive documents were retrieved from Element Zero's patent attorney, not the Respondents. These matters indicate to me a reason to doubt the adequacy of the Respondents' discovery in respect of Category 13, because relevant patents have been overlooked, and the Respondents have sourced responsive documents otherwise than from their own records.
- 31. At paragraph 81 of Dunn 7, Ms Dunn refers to the 'Ore composition after drying' document and asserts it "is of a type that is publicly available". In that paragraph, she refers to a document "Fortescue Ore Reserves and Mineral Resources Update: Operating Properties" (Update Document). However, I am informed by Dr Anand Bhatt, and believe, that the Update Document does not disclose the data concerning Fortescue's blend compositions that is contained in the 'Ore composition after drying' document, which Fortescue provides to its customers. Rather, the Update Document (pp 10-13) sets out data regarding ores from individual Fortescue mine sites, and an iron 'cut-off value' (i.e., a threshold value for iron content) for individual mines. Further, I am

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informed by Adrian Huber, Legal Manager at Fortescue, and believe, that Fortescue's blend composition data is confidential information to Fortescue.

Conclusion

- 32. In light of the discovery deficiencies notified to the Respondents to date, the fact that additional documents have been produced by the Respondents that are responsive to the discovery categories, the further deficiencies in the EZ Respondents' approach to discovery apparent from Dunn 7 and addressed above, I consider there is reason to doubt the adequacy of the Respondents' discovery across all of the categories ordered by the Court.
- 33. The Applicants will be severely prejudiced unless the proceeding is adequately and properly prepared for trial. I am concerned that unless the Applicants are granted orders for access and inspection of the materials the subject of the search order dated 14 May 2024, that the proceeding will not be adequately and properly prepared for trial. In particular, I am concerned that in view of the reasons to doubt the adequacy of the Respondents' discovery that evidence will not be available to the Court that is necessary to adequately and properly determine the issues in the proceeding which will prejudice the Applicants.

Affirmed by Paul Alexander Dewar at London in the United Kingdom on 18 September 2025
Before me:

Signature of deponent

Signature of witness

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Grace Louise O'Connor Level 28, 500 Bourke Street, Melbourne, Victoria 3000 An Australian Legal Practitioner (within the meaning of the *Legal Profession Uniform Law* (*Victoria*))

A person authorised under section 19(1) of the Oaths and Affirmations Act 2018 to take an affidavit This affidavit was sworn by the deponent by audio visual link using an electronic copy of the affidavit and not the original in completing the jurat requirements.

No. NSD527 of 2024

Federal Court of Australia

District Registry: New South Wales

Division: General

FORTESCUE LIMITED (ACN 002 594 872) and others

Applicants

ELEMENT ZERO PTY LIMITED (ACN 664 342 081) and others

Respondents

ANNEXURE COVER SHEET

This is **Annexure PAD-77** produced and shown to **PAUL ALEXANDER DEWAR** at the time of affirming his affidavit.

Before me:

Jessica Sapountsis

From: Jessica Sapountsis

Sent: Tuesday, 16 September 2025 2:20 PM

To: Caitlin Meade

Cc: Paul Dewar; Grace O'Connor; Rebecca Dunn; Rohit Dighe; Kevin Huang; Oliver

Hoare; Daisy Cullen; Amelia Cooper; Mike Hales; Daniella Lambert; Michael Williams

Subject: RE: Fortescue Limited & Ors v Element Zero Pty Ltd & Ors - Federal Court

Proceedings NSD 527/2024 [ITUSEONLY-LAW.FID86345]

Dear Colleagues

We refer to your clients' subsequent production of documents on 11 September. In our email of 10 September 2025, we requested that the metadata for those documents includes an indication of the category/categories each document is responsive to, but that information was not included in the folder you sent through on 11 September, nor provided separately. Could you please provide us with an indication of:

- 1. which are the 490 documents that have been produced on the basis that they were produced by the Third Respondent in relation to categories 11(d) and 11(f); and
- 2. for the remaining 75 documents, which particular documents are responsive to each of the deficiencies identified in your 9 September 2025 letter (ie, Items 2, 376-378, 379, 380, 381, 384, 387, 396-398, 399, 407, 408, 425, 426, 428-429, 430, 448, 493 and 494). That is, which documents are said to be responsive to Item 2, which documents are said to be responsive to Items 376-378, etc.

Without the above information, we are not in a position to consider whether the EZ Respondents have satisfactorily responded to the deficiencies identified in relation to the above Item numbers.

Finally, we note your indication in your 9 September letter that the documents have been provided on the basis of the confidentiality undertakings provided by Fortescue's external counsel, but the confidentiality status of the documents has not been coded in Relativity. Can you please confirm whether or not your clients' claim confidentiality in respect of every one of these documents?

Yours sincerely

Jessica Sapountsis

Managing Associate



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We extend our respect to all Aboriginal and Torres Strait Islander peoples throughout Australia and acknowledge the Traditional Owners and Custodians of the lands on which we work. We recognise their ongoing connection to land, sea and community.

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No. NSD527 of 2024

Federal Court of Australia

District Registry: New South Wales

Division: General

FORTESCUE LIMITED (ACN 002 594 872) and others

Applicants

ELEMENT ZERO PTY LIMITED (ACN 664 342 081) and others

Respondents

ANNEXURE COVER SHEET

This is **Annexure PAD-78** produced and shown to **PAUL ALEXANDER DEWAR** at the time of affirming his affidavit.

Before me:

6 (04/2023 15:58 [MZ001 Overview - Final (V9) pptx 22/02/2023 17:16 [Palgycound questions, 620.20] 17/02/2024 14:26 [In oxide Chen, 2021 J. Electrochem, 50c. 168 112509 pdf 20/01/2023 12:05 [10182-0000 CS-CTC-0001 Standard Terms and Conditions - Element Zero Final Rev B.pdf 26/07/2023 7:27 [Rely Regional questions 25023 alocx	BWJ.5000.0005.0200 BWJ.5000.0004.5383 BWJ.5000.0004.5428 BWJ.5000.0004.4325
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20/06/2023 18:14 Lab scale Bleed_leaching_EW process TBA 20_6_23.pdf	BWJ.5000.0005.7429; BWJ.5000.0005.7453
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No. NSD527 of 2024

Federal Court of Australia

District Registry: New South Wales

Division: General

FORTESCUE LIMITED (ACN 002 594 872) and others

Applicants

ELEMENT ZERO PTY LIMITED (ACN 664 342 081) and others

Respondents

ANNEXURE COVER SHEET

This is **Annexure PAD-80** produced and shown to **PAUL ALEXANDER DEWAR** at the time of affirming his affidavit.

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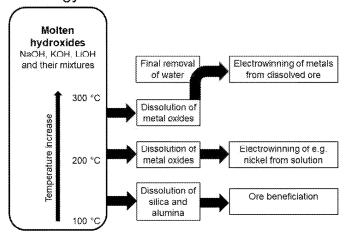
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(54) Title: METHOD OF ORE PROCESSING

FIGURE 1

Technology base



(57) **Abstract:** The invention relates to a method of providing an ore concentrate solution suitable for beneficiation processing, the method including the step of contacting an ore with one or more metal bases at elevated temperature. The one or more metal bases at elevated temperature may form a super-alkaline media that partially or fully dissolves the ore. Typically, the one or more metal bases are alkali metal bases, preferably chosen from lithium hydroxide, sodium hydroxide, potassium hydroxide, rubidium hydroxide or caesium hydroxide, or alkaline earth bases, preferably chosen from calcium hydroxide, barium hydroxide or strontium hydroxide. The one or more metal bases at elevated temperature may form a super-alkaline media that partially or fully dissolves the ore.

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METHOD OF ORE PROCESSING

FIELD OF INVENTION

[0001] The present invention relates to the field of ore processing.

[0002] In one form, the invention relates to processing ores to provide valuable products such as metals, metal compounds, metalloids or intermediate compounds suitable for further down-stream processing.

[0003] In another aspect the present invention is suitable for providing processed ore for down-stream beneficiation processes.

[0004] In one particular aspect the present invention is suitable for providing processed ore for down-stream electrometallurgical processes such as electrodeposition or electrowinning.

[0005] It will be convenient to hereinafter describe the invention in relation to iron ore, however it should be appreciated that the present invention is not so limited but extends to a wide range of ores, and a wide range of valuable products including products based on metals and metalloids. Furthermore, it will also be convenient to describe the invention in relation to provision of processed material for electrometallurgy but can be extended to other forms of extractive metallurgy and ore beneficiation processes.

BACKGROUND ART

[0006] It is to be appreciated that any discussion of documents, devices, acts or knowledge in this specification is included to explain the context of the present invention. Further, the discussion throughout this specification comes about due to the realisation of the inventor and/or the identification of certain related art problems by the inventor. Moreover, any discussion of material such as documents, devices, acts or knowledge in this specification is included to explain the context of the invention in terms of the inventor's knowledge and experience and, accordingly, any such discussion should not be taken as an admission that any of the material forms part of the prior art base or the

common general knowledge in the relevant art in Australia, or elsewhere, on or before the priority date of the disclosure and claims herein.

[0007] The term 'crude ores' refers to ores, metals, metalloids, minerals, and other products containing mineral substances that are mined or otherwise removed from the ground and may be sized or crushed. Crude ores are subjected to further treatment or concentrating as part of industrial processing to separate valuable minerals from waste rock or gangue. "Beneficiation' is a term used to refer to any treatment that improves or benefits the economic value of ore, to provide a higher grade product (called an 'ore concentrate') and a waste stream.

[0008] The further treatment typically includes extractive metallurgy to remove metals from natural mineral deposits. Extractive metallurgy techniques are commonly grouped into four categories: hydrometallurgy, pyrometallurgy, ionometallurgy and electrometallurgy.

[0009] Electrometallurgy involves metallurgical processes carried out in some form of electrolytic cell. The most common types of electrometallurgical processes are electrorefining and electrowinning.

[0010] Electrowinning is an electrolysis process used to recover metals from aqueous solution, usually after an ore has undergone one or more hydrometallurgical processes. An electrical current is passed from an inert anode through a leach solution containing the dissolved metal ions so that the metal is deposited onto a cathode and recovered.

[0011] Electrorefining uses a similar process to remove impurities from a metal. In electrorefining, the anode consists of the impure metal to be refined. The impure metallic anode is oxidized and the metal dissolves into solution. The metal ions migrate through the acidic electrolyte towards the cathode where the pure metal is deposited.

[0012] Ionometallurgy uses ionic liquid or eutectic melts to extract and or convert metals and minerals.

[0013] Apart from metals, many other commercially valuable products are derived from ores. For example, silica is an abundant, and chemically complex material found in several minerals, particularly quartz. It is extremely valuable to the microelectronics, food, and pharmaceutical industries.

[0014] The mining industry is constantly seeking 'green' ore processing technology to reduce emissions and waste.

[0015] For example, the steel industry accounts for about 7% of global carbon dioxide emissions and reducing carbon pollution from iron ore processing is important in efforts to avoid further climate change. Accordingly, in recent times there has been a focus on developing green iron – a higher value form of iron that has been stripped of impurities to leave purer iron without using processing that creates carbon dioxide emission. There is a worldwide effort to reduce carbon emissions by employing hydrogen-based steelmaking using blast furnaces or direct reduced iron plants followed by electric arc furnaces to replace fossil fuels but requires pellets of high-grade iron ore with low impurity content.

[0016] A limited quantity of high-grade ore is currently mined, mostly in the Americas, Europe and the Middle East. As supplies of high-grade ore are mined-out, it will be necessary to exploit lower-grade ores. Countries which already mine lower-grade ores, will need to further refine the crude product to make it suitable for reduction with hydrogen in blast furnaces or direct reduced iron plants in order to compete with suppliers of higher grade ore and to meet the expectations of overseas ore processors.

[0017] Australia has for many years relied on direct shipping ore – that is, ore that can be simply dug up and exported without further processing or with very limited processing, (such as blending or drying). The three main types of Australian iron ore are hematite, goethite and magnetite. Crude hematite/goethite is higher grade, and deposits are dwindling while magnetite deposits are large and comparatively low grade – but can be used to produce very high-grade concentrates.

[0018] Iron ore mining is a high-volume, low-margin business. It is capital intensive and requires substantial investment in infrastructure. Producers must get the best return from their product and the return depends heavily on iron ore grade and demand. Over

the last 10 years, the premiums for high grade ore and discounts for low grade ore have increased, causing steelmakers to demand higher grade ore with less impurities.

[0019] The grade of Australian iron ore has declined over recent years and miners are experiencing significant depletion of their reserve deposits. In order to compete with other iron ore producers Australia must develop options for production of higher grade iron ore or its derivatives.

[0020] Various pathways have been evaluated to produce green iron, including electrochemically converting iron ore to iron at a wide range of temperatures (60 to 2,000 °C) without coal, natural gas, or other reductants, and using green hydrogen as green reductant to replace fossil fuel based reductants in blast furnaces of DRI plants. However, it has proved difficult to make green processes economically viable and efficient. Green pyrometallurgical processes also rely on a consistent, uninterrupted source of sufficient power, but this can be difficult to supply in remote mining areas where crude ore processing is carried out due to highly intermittent nature of wind and solar power generation.

SUMMARY OF INVENTION

[0021] An object of the present invention is to provide a method of conversion and extraction of ores.

[0022] Another object of the present invention is to provide a green process, or at least a process that facilitates green processing of ores.

[0023] A further object of the present invention is to alleviate at least one disadvantage associated with the related art.

[0024] It is an object of the embodiments described herein to overcome or alleviate at least one of the above noted drawbacks of related art systems or to at least provide a useful alternative to related art systems.

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[0025] In a first aspect of embodiments described herein there is provided a method of producing an ore concentrate solution, the method including the step of contacting ore with one or more metal bases, preferably alkali metal or alkaline earth bases, at elevated temperature. Preferably the ore concentrate solution is suitable for downstream beneficiation processing, such as extractive metallurgy.

[0026] The ore fed into the process of the present invention is typically a crude ore but may be an ore that has undergone some refinement to become a concentrated ore. Once the ore has been treated according to the method of the present invention to provide an ore concentrate solution, it can readily be supplied to downstream processes, such as electrometallurgical extraction processes. Thus, it is possible to avoid traditional concentration processes such as flotation, electrostatic separation, or magnetic separation or dewatering that are inefficient in terms of energy consumption.

[0027] Typically, ore for use in the present invention is any crude ore, or ore concentrate comprising a metal or metaloid. Preferably the ore is chosen from the group comprising one or more of the following: iron ore including hematite, goethite, magnetite, titanomagnetite and pisolitic ironstone; aluminium containing ores including bauxite, cryolite and corundum; gold ores including gold-polysulfide, gold-quartz, gold-telluride, gold-tetradymite, gold-antimony, gold-bismuth-sulfosalt, gold-pyrrhotite, and gold-fahlore; manganese containing ores such as romanechite, manganite hausmannite and rhodochrosite; lead ores including galena, cerrusite and anglesite; zinc ores including calamine and smithsonite; cobalt containing ores; uranium containing ores; copper containing ores including copper pyrite, chalcopyrite, bornite, covellite, chalcocite, malachite, cuprite and copper glance; nickel containing ores such as laterites and magmatic sulphide deposits; silver containing ores such as argentite; tin containing ores such as cassiterite, tinstone, stannite or cylindrite; and quartz. In a particularly preferred embodiment the ore is iron ore, which is particularly rich in iron oxides, particularly in the form of magnetite (Fe₃O₄), hematite (Fe₂O₃), goethite (FeO(OH)), limonite (FeO(OH).n(H₂O)) or siderite (FeCO₃).

[0028] In another preferred embodiment the ore is an ore concentrate that includes species such as nickel oxide, nickel hydroxide or nickel sulphide.

[0029] In a further preferred embodiment the ore is an ore concentrate that includes species such as copper sulphide or copper-iron sulphide.

[0030] The metal base or bases at elevated temperature comprise a super-alkaline media. Upon contact with the metal base, the ore fully or partially dissolves and/or metal containing moieties are chemically converted to solvable species. Without wishing to be bound by theory it is believed that sulphide ores, for example, are converted to oxides.

[0031] Additional compounds may facilitate the dissolution or chemical conversion of the ore. In particular, the addition of silicates may promote dissolution or chemical conversion of the ore, particularly ore concentrates.

[0032] Alkali metal or alkaline earth bases suitable for use in the present invention are preferably hydroxides, although other bases such as metal oxides or metal ammonium species may also be used.

[0033] Typically, the metal base is chosen from alkali metal bases such as lithium, sodium, potassium, rubidium or caesium hydroxide, or alkaline earth bases such as calcium, barium or strontium metal hydroxides. In a particularly preferred embodiment, the metal base is chosen from lithium hydroxide, sodium hydroxide, potassium hydroxide or calcium hydroxide. In a particularly preferred embodiment, the super-alkaline media comprises 45 wt% to 100 wt% sodium hydroxide and/or potassium hydroxide.

[0034] One or more metal bases may be contacted with the ore, and combinations of metal bases may be in the form of a eutectic mixture. Eutectic mixtures of sodium, potassium and/or lithium hydroxide is particularly preferred. In some embodiments, for economic reasons NaOH is preferred but pure NaOH may not be as efficient as the combination of NaOH with KOH to form a eutectic system.

[0035] The super-alkaline media comprising alkali metal or alkaline earth bases are contacted with the ore at elevated temperature, preferably a temperature above 160 °C, or above 200 °C, preferably above 250 °C, more preferably above 300 °C. In a particularly preferred embodiment, the alkali metal or alkaline earth bases are contacted with the ore

at a temperature of 160 °C to 400 °C, preferably 200 °C to 350 °C, more preferably 250 °C to 350 °C.

[0036] For example, with reference to eutectics, most mixtures of NaOH and KOH have lower melting points than the constituent compounds. For a 1:1 molar ratio of NaOH:KOH, the eutectic forms at 170 °C. If adsorbed or crystalline water is present, such as in a 1:1:1 ratio of NaOH:KOH:H₂O, the temperature of formation of the eutectic can be below 100 °C.

[0037] Once the super-alkaline media is contacted with the ore and the ore is partially or fully dissolved, the combination may be cooled to form a solid and then re-heated for further processing.

[0038] Molten metal bases, particularly hydroxides often include impurities such as water. Preferably metal bases incorporated in the super-alkaline media of the present invention will include water in amounts of no more than one mole of water per more of hydroxide. It is also possible to drive off water from the super-alkaline media by short term heating of the super-alkaline media to higher temperatures (i.e., > 450 °C). A shield of inert gas over the super-alkaline media can then be used to restrict or prevent reabsorption of water.

[0039] The metal bases used in the present invention may include small amounts of chemical impurities. For example, sodium hydroxide may form, or include small amounts of sodium carbonate (Na₂(CO₃)).

[0040] In a second aspect of embodiments described herein there is provided a method of refining an ore, the method including the steps of:

- (i) contacting the ore with one or more metal bases, preferably alkali metal or alkaline earth bases, at elevated temperature;
- (ii) feeding a solution formed in step (i) to a beneficiation process.

[0041] In one aspect of the present invention, the beneficiation process may be, for example, an extraction process for removing silica and/or alumina or other impurities including titania, phosphorus and manganese.

[0042] In another aspect of the present invention, the beneficiation process may be, for example, an extractive metallurgical process, preferably electrometallurgy, to deposit metal from the solution.

[0043] Upon contact of the ore with one or more metal bases, the ore may fully or partially dissolve or chemically convert moieties in the ore to solvable species. While ultimately the solution thus formed may be fed to a downstream extractive metallurgical process such as an electrochemical process for selective electrodeposition of target metals, it may be advantageous to include a step facilitating extraction of particular elements, such as nickel, cobalt, molybdenum, lithium, aluminium and silicon. The solution may be subjected to further beneficiation processes.

[0044] Therefore, in a third aspect of embodiments described herein there is provided a method of concentrating an ore, the method including the steps of:

- (i) contacting the ore with one or more metal bases, preferably alkali metal or alkaline earth bases, at elevated temperature to form an ore concentrate solution;
- (ii) extracting one or more components of the ore from the ore concentrate solution;

and optionally,

(iii) feeding the extracted solution to an extractive metallurgical process, preferably electrometallurgy, to deposit metal;

and optionally,

(iv) passing the extracted ore concentrate solution to further beneficiation processes.

[0045] In a fourth aspect of embodiments described herein there is provided a method of concentrating an ore, the method including the steps of:

- (i) contacting the ore with one or more metal bases, preferably alkali metal or alkaline earth bases, at elevated temperature,
- (ii) converting sulphide species in the ore to oxides in solution;
- (iii) extracting one or more of the oxides from the solution; and optionally,
- (iii) passing the extracted oxides to further refining processes.

[0046] In a fifth aspect of embodiments described herein there is provided a method of concentrating an ore, the method including the steps of:

- (i) contacting the ore with one or more metal bases, preferably alkali metal or alkaline earth bases, at elevated temperature;
- (ii) forming a solution comprising dissolved components from the ore:
- (iii) removing the components from the solution; and optionally
- (iv) forwarding the purified solution from step (iii) to a downstream beneficiation process.

[0047] Components removed from the solution may include aluminium and silicate species. The components removed from the solution may be converted into valuable commercial products such as geo-polymers (inorganic aluminosilicate polymers) or zeolites (generally expressed as Mⁿ⁺1/2(AlO₂)⁻ (SiO₂)x.yH₂O where Mⁿ⁺1/2 is a metal ion, typically Na⁺, K⁺, Ca²⁺, Mg²⁺, or H⁺.

[0048] In another aspect, the present invention provides a concentrated ore, or a refined ore, produced according to the method of the present invention.

[0049] In yet another aspect, the present invention provides commercial products produced according to the method of the present invention. In a particularly preferred embodiment the commercial product is a metal.

[0050] Other aspects and preferred forms are disclosed in the specification and/or defined in the appended claims, forming a part of the description of the invention.

[0051] In essence, embodiments of the present invention stem from the realization that a super-alkaline media can be used to convert ore, including crude ore, into useful concentrated ores or commercially valuable chemical products.

[0052] Advantages provided by the process of the present invention comprise the following:

- can be carried out at ambient pressure;
- low energy consumption;

- high efficiency conversion of ores to valuable chemical species;
- high efficiency refinement of ores, particularly crude ores;
- provision of an ore feedstock to further downstream ore refining processes; and
- provision of chemical species suitable for processing into valuable chemical products.

[0053] Further scope of applicability of embodiments of the present invention will become apparent from the detailed description given hereinafter. However, it should be understood that the detailed description and specific examples, while indicating preferred embodiments of the invention, are given by way of illustration only, since various changes and modifications within the spirit and scope of the disclosure herein will become apparent to those skilled in the art from this detailed description.

BRIEF DESCRIPTION OF THE DRAWINGS

[0054] Further disclosure, objects, advantages and aspects of preferred and other embodiments of the present application may be better understood by those skilled in the relevant art by reference to the following description of embodiments taken in conjunction with the accompanying drawings, which are given by way of illustration only, and thus are not limitative of the disclosure herein, and in which:

<u>Figure 1</u> illustrates the use of molten hydroxides as a super alkaline media at different temperatures.

<u>Figure 2</u> is a plot illustrating deposition of iron from a solution formed by contacting haematite ore with an NaOH/KOH eutectic at five temperatures – 250 °C, 275 °C, 300 °C, 325 °C and 350 °C. The plot records current (I) in mA against voltage (E) in volts.

<u>Figure 3</u> is a plot illustrating the effect of removing water from the super-alkaline media. The plot records current (I) in mA against voltage (E) in volts.

DETAILED DESCRIPTION

[0055] The present invention provides a method of refining ore, such as crude ore or concentrated ore, for downstream processing, particularly extractive metallurgy. The method includes the step of contacting ore with one or more metal bases, preferably a super alkaline media formed from alkali metal and/or alkaline earth bases, at elevated temperature. Where two or more alkali metal and/or alkaline earth bases are used, typically the super alkaline media will be in the form of a eutectic.

[0056] Figure 1 illustrates the use of molten hydroxides as a super alkaline media at different temperatures. At lower temperatures, around 100 °C, the super alkaline media is particularly useful for dissolving species such as silica and alumina from ore. The concentrated ore may be passed on to other beneficiation processes, and the alumina or silica used as valuable products. At medium temperatures, around 200 °C, the super alkaline media is particularly useful for dissolution of certain metal oxides which can be recovered by subjecting the solution to electrowinning processes. At higher temperatures, around 300 °C, the super alkaline media is particularly useful for dissolving metal oxides such as iron which can be recovered by subjecting the concentrated ore to electrowinning processes.

[0057] In particular, the super alkaline media can be used to:

- Fully or partially dissolve or chemically convert metal containing ore to solvable species followed by electrochemical deposition of metal from this solution;
- Fully or partially dissolve or chemically convert ore to solvable species facilitating
 extraction of particular elements (such as nickel, cobalt, molybdenum, aluminium,
 lithium, and silicon) followed by selective electrodeposition of the element from the
 molten hydroxide or followed by chemical processing of the dissolved element;
- Fully or partially convert sulphide ore or concentrate (such as iron or nickel) to oxides followed by conventional processing of these oxides (chemical and/or electrochemical but not necessarily in the molten hydroxide);
- Partially dissolve mineral ores to remove components (such as alumina and silica)
 followed by down-stream processing of the purified ore and with the possibility of

conversion of the removed components into commercial products like geo-polymers or zeolites.

[0058] Ore Concentrates

[0059] For any of the abovementioned processes, additional compounds may be used to facilitate the dissolution of the ore or chemical conversion of components into soluble species. In particular, the addition of silicates may enhance the conversion of the solid oxide into metal-silicates that form a solution with the super-alkaline media. For example, the addition of silicates such as quartz, feldspar, mica, amphibole, pyroxene, olivine or aluminium-silicate may be advantageous, particularly for ore concentrates.

[0060] When ore concentrates are exposed to molten hydroxides initially, they may form a solid metal oxide. For example, nickel hydroxide or nickel sulphide concentrates may initially form a solid iron-nickel oxide. This solid oxide may also contain small amounts of other elements such as iron, manganese, magnesium, copper and cobalt (but very little silicate).

[0061] By controlling the process parameters such as temperature and silica concentration it is possible to perform selective dissolution of metals contained in the solid oxide. Undesirable oxides can remain undissolved in the solid oxide, controlling the purity or species in the solution.

[0062] Metals may be electro deposited directly from the solution or be isolated by conventional means in a down-stream process (for example, neutralization followed by conventional electrowinning).

[0063] For sulphide concentrates this procedure is particularly advantageous because it eliminates the need for the conventional high temperature 'roasting' process.

EXAMPLES

[0064] The present invention will be further described with reference to the following non-limiting examples:

[0065] Example 1 – Haematite

[0066] The method of the present invention was applied to a sample of iron ore "dust" from Western Australia, comprising approximately 24 wt% Si, approximately 21 wt% iron and 1 wt% Ni.

[0067] A super-alkaline media was formed, comprising a eutectic of NaOH/KOH in 1:1 molar ratio at 200 °C. No attempt to remove water from the eutectic was performed and it is known that the equilibrium water content at this temperature is 8 to 10 wt% depending on NaOH:KOH ratio. Upon contact of the super-alkaline media with the iron ore dust, a solution formed comprising aluminates, silicates, and some iron oxides (magnetite, haematite, goethite, limonite, and siderite). However, the majority of iron oxides remain in the solid state at 200 °C. The concentration of iron oxide species in the solution increased with increasing temperature and thereby decreasing water content at atmospheric pressure, reaching full dissolution at 350 °C.

[0068] Haematite was recovered from the solution at 200 °C by simple decanting of the liquid followed by rinsing with water. Analysis indicated that the haematite to have 93 wt% purity (61% iron), which is considered 'export grade'.

[0069] Example 2 - Nickel

[0070] The solution formed in Example 1 was maintained at 200 °C and atmospheric pressure and fed an electrolysis cell in an electrowinning process. The solution comprised nickel originating from the approximately 1% nickel in the original ore.

[0071] An electric current was passed from an inert gold anode, through the solution and nickel and iron were deposited onto the cathode in an electroplating process in a ratio of 7:1 (Ni:Fe).

[0072] Example 3 – Ore Dissolution in Molten Hydroxide

[0073] A super-alkaline media was formed, comprising a NaOH/KOH (1:1 molar ratio) eutectic at 300 °C. Haematite ore was progressively added and a solution formed. At low concentrations of haematite, the solution was light green, the colour becoming more intense. At a concentration of 200 g of haematite in one litre of the super-alkaline media at 300 °C, the solution was an intense green/black colour.

[0074] Ores of different quality having Fe content from 48% to 62% Fe were similarly tested and all showed the same dissolution behaviour.

[0075] The resulting solution exhibited low viscosity (appearing to behave similarly to water) above 250 °C which is particularly suitable for electrochemical processing. The solution was cooled to 25 °C, at which temperature it became solid. The solid material could be re-melted and subjected to further metallurgical extraction.

[0076] At higher concentrations of haematite ore in the super-alkaline media, a solution formed in the precipitate. When the solution was cooled to 25 °C and then remelted, the precipitate remained an amorphous solid that would not dissolve or melt when subjected to temperatures up to 300 °C.

[0077] Both haematite and goethite are iron oxides in which iron is in oxidation state (III), imparting the characteristic red 'rust' to red-brown colour to the ore. The development of a distinctive green colour indicates that at least some of the iron(III) has changed oxidation state during the formation of the solution. Without wishing to be bound by theory the green colour of the solution could possibly be due to:

- formation of soluble iron(II) silicates; and/or
- formation of 'green rust' comprising mixed valence (oxy)hydroxides having iron in mixed oxidation states (+2 and +3). Low melting point iron silicates exist in both Fe(II) and Fe(III) species, with only the Fe(II) silicate being green; and/or
- formation of other iron compounds in solution.

[0078] A few possible reaction schemes support the observed conversion to Fe(II), all of which suggest the generation of oxygen, which is consistent with the observation of bubbles when the super-alkaline media contacts the ore according to the following:

- $2Fe_2O_3 + 4NaOH \rightarrow 4(Na^+, HFeO_2^-) + O_2$
- Fe₂O₃ + 2NaOH → H₂O + O₂ + 2FeO + 2Na (The sodium would react with water and form NaOH and H₂; FeO is soluble in alkaline media.)
- 2Fe₂O₃ → 4FeO + O₂
- $2Fe_2O_3 + 2H_2O \rightarrow 4FeOOH$, followed by $4FeOOH + 2H_2O \rightarrow 4Fe(OH)_2 + O_2$

[0079] The generation of oxygen could be useful for novel applications such as the mining or refining of minerals in anaerobic or oxygen deficient atmospheres. This could facilitate mining ore resources on the moon, on asteroids or planets, such as Mars.

[0080] Example 4 – Influence of Temperature on Iron Deposition

[0081] The solution of Example 3 was separately fed into an electrolysis cell in an electrowinning process kept at atmospheric pressure and deposition was carried out in the cell at temperatures of 250 °C, 275 °C, 300 °C, 325 °C and 350 °C respectively.

[0082] An electric current was passed from an inert gold anode, through the solution and iron was deposited onto an iron cathode in an electroplating process. It was noted that during the deposition process some parasitic hydrogen evolution also occurred.

[0083] Figure 2 is a plot illustrating the results of the iron deposition from each solution. The plot illustrates successful deposition at 250 °C, and that deposition rate increases with increasing temperature. The increase in current with increase in temperature accounts for both decrease in solution viscosity and normal activity increase.

[0084] Example 5 – Influence of Water in the Super-Alkaline Media

[0085] Many alkali metal or alkaline earth bases are hygroscopic. Hydroxides in particular are very hygroscopic and even 'pure' commercially available hydroxides often contain up to 10 wt% water at 200 °C.

[0086] The presence of water in electrochemical processing such as electrodeposition can lead to parasitic hydrogen evolution, due to splitting of water. This side reaction reduces the overall efficiency of the electrodeposition reaction. Removal of water is important in metal deposition, particularly iron deposition as the thermodynamic reduction potentials for water and iron oxides favour hydrogen evolution over iron deposition.

[0087] Water can be effectively driven off by short term heating of the super-alkaline media to higher temperatures (i.e., > 450 °C). A shield of inert gas should then be maintained over the solution during deposition to restrict or prevent reabsorption of water. Prolonged heating of the solution to >350 °C during deposition also removes enough water to substantially reduced hydrogen evolution.

[0088] Figure 3 is a graph illustrating the effect of removing water from the superalkaline media. The graph records current (I) in mA against voltage (E) in volts. The first plot was made at 12.00 pm, the second at 14.00 pm the third at 16.00 pm and the final plot at 16.30 pm. The increase in slope of the 16.00 pm graphs at about –2.2 Volts occurs as water is driven out of the super alkaline media. Overall, the graph illustrates prolonged heating at 350 °C successfully drives off water, but the process is quite slow. The plot also reflects the 'real' iron deposition rate.

[0089] Example 6 – Dissolution/Conversion of Iron Ore as a Function of Impurities

[0090] Three dried hematite ore samples (dried at 200 °C for 2 hours) having the same Pilbara origin but with different iron content (55%, 60% and 62% Fe, respectively) were reacted with molten hydroxide in three Teflon lined containers.

[0091] A 20 g portion of each ore sample was added to 48 g of 1:1 (molar) NaOH:KOH at 310 °C and stirred for one minute and dark green solutions developed. The temperature was kept at 310 °C for four hours and the samples were then allowed to cool to room temperature under Teflon lids.

[0092] To determine the dissolution/conversion of ore, the Teflon liners with the ore/hydroxide samples were immersed in 150 ml of 5.5 M HCl and allowed to react for 24 hours with agitation. The liquor was then decanted off and 50 ml for fresh 5.5 M HCl was added and allowed to react for 2 hours and decanted again. The unreacted ore was then washed three times in distilled water and dried in air at 150 °C before weighing. The characteristics of each sample are listed in Table 1.

[0093] Table 1:

Fe in ore	62%	60%	55%	HCI control
Fe ₂ O ₃ in ore	89%	86%	70%	
Solid after test (g)	10.9	9.972	7.822	19.588
Dissolved in test (g)	9.1	10.028	12.178	0.412
wt% ore in solution	19.0	20.9	25.4	

[0094] It can be seen from Table 1 that the saturation of dissolved ore in molten hydroxide increases with the increased quantity of impurities in the ore. This is not unexpected as the main impurities — silica and alumina — are well-known to readily dissolve under alkaline conditions. However, the increase in dissolved ore is larger than the additional quantity of impurities going from 62% to 60% and to 55% Fe content. This suggests that the impurities are aiding the dissolution of the iron oxides in molten hydroxides.

[0095] Two control experiments were conducted using the 60% Fe ore sample. In one experiment the reaction time at 310 °C was decreased from four to one hour. This did not result in a change in the measured saturation of dissolved ore, which strongly suggests that the reaction/conversion time is well below one hour under the conditions used.

[0096] In a separate experiment, 20 g of 60% Fe ore was allowed to react with 200 ml of 5.5 M HCl for 26 hours. The undissolved ore was rinsed in water, dried, and weighed. The exposure to HCl only caused a very minor decrease in weight (see Table 1), meaning that the effect seen with exposure to molten hydroxide is caused by the hydroxide, not by the titration with HCl.

[0097] Example 7 – Dissolution of Manganese(IV) Oxide (MnO₂)

[0098] Manganese oxide is a common impurity in Australian iron ore, usually limited to a range of up to about 1%. The manganese impurities are rarely part of the iron oxide lattice, but rather occur as well-defined grains of MnO₂. The low level of manganese oxides in the ore makes it difficult to determine what happens to it during the dissolution process of the (iron) ore. To address this, 5 wt% of synthetic MnO₂ was added to 1:1 (molar) NaOH:KOH at 300 °C. The molten hydroxide immediately turned black and gradually shifted to dark green/black after 24 hours without precipitates. Based on the colour observed at that temperature, it is unlikely that manganese(II) hydroxide formed because it is white and decomposes at ~140 °C. Instead, the green colour indicates partial formation of the manganate(VI) ion.

[0099] Electrodeposition was attempted from the molten hydroxide solution at 300 °C under conditions known to produce iron from dissolved iron ore. Nickel foil was used as both anode and cathode material and a voltage of 1.8 V was applied between the electrodes. A bright green solution was formed around the anode, indicating formation of manganate as the oxidized product. The deposits on the cathode were analysed and found to be a mixture of manganese oxides, with the manganese mainly in oxidation states +2 and +4.

[00100] This experiment shows that metallic manganese is not likely to be deposited as the cathode product (pure or as an alloying element) under the conditions used when dissolved iron species are electrodeposited. Instead, trace impurities of manganese oxides are to be expected.

[00101] <u>Example 8 – Dissolution of Molten Hydroxide Followed by Separation at High Temperatures</u>

[00102] Sodium hydroxide (250 g) was allowed to melt at 335 °C in a heated lab scale thickener and 25 g of dried (400 °C) iron ore powder was added. After one hour when the ore was dissolved, the temperature was raised to 370 °C and kept at this temperature for one hour. The elevated temperature caused the dissolved iron species to de-hydrolyse

and thereby phase separate from the bulk of molten hydroxide in the conical part of the thickener. This allowed the iron-rich intermediate to be removed from the thickener by gravimetric means.

[00103] The main impurities from the ore (silica and alumina) remained in solution in the molten hydroxide as silicates and aluminates. The process could be repeated by lowering the temperature to 335 °C, adding more iron ore, raising the temperature again to 370 °C causing more iron rich intermediate to separate out.

[00104] The iron rich intermediate was added to a eutectic melt of sodium and potassium hydroxide and electrodeposition was carried out at temperatures in the 220 °C to 310 °C range.

[00105] The dissolution and separation can also be performed directly in the eutectic melt at high temperatures. However, this incurs additional cost because the more expensive potassium hydroxide is spent in the process by conversion to potassium silicates and potassium aluminates.

[00106] Example 9 – Dissolution of Iron Ore in Molten Hydroxide Eutectics Followed by Removal of Impurities from the Surface

[00107] Four kilograms of hydroxide eutectic (1:1 NaOH:KOH by weight) was heated in a vertical kiln having a nickel liner to 300 °C. A sample of 400 g of dried iron ore (hematite, 55% Fe) was added and stirred for 30 minutes until all the iron ore was dissolved. The stirring was then switched off and the temperature was gradually and slowly decreased.

[00108] At temperatures close to the freezing point of the mixture (~230 °C to 210 °C), a liquid-liquid gravimetric phase separation occurred. The bottom phase contained the heavier, iron-rich species and the upper layer contained the impurity rich species (mainly silicates and aluminates). The impurity rich phase could be removed by carefully pumping the phase at the upper level of the solution.

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[00109] When the temperature was further lowered (to 200 °C) a white crust formed on the surface of the solution, which was removed by simple mechanical means. Elemental analysis revealed that this crust contains up to 50% impurities in a hydroxide matrix.

[00110] These methods of separation of impurities in liquid or in solid form from the dissolved ore both provide practical methods for beneficiation prior to further processing of the iron-rich solution, for example, by electrowinning.

[00111] Example 10 - Magnetite Concentrate

[00112] A sample of 10 wt% of magnetite concentrate (67% iron, 5% silica, \sim 50 μ m particle size) was added to a mixture of molten NaOH and KOH (3:1 molar concentration) at 310 °C. The magnetite concentrate dissolved quickly under stirring, forming a black-brown solution. This solution was used without further treatment for electrowinning of iron at 310 °C.

[00113] The experiment was repeated with a 1:1 (molar) ratio of NaOH:KOH and the magnetite concentrate was dissolved at 310 °C. After the dissolution was completed, the temperature was lowered to 240 °C before electrowinning of iron was performed.

[00114] Example 11 - Silica, Silicates and Quartz

[00115] Silica and other variations of the SiO₂ motif are known to easily dissolve in alkaline media and this is a significant aspect of various, established routes of industrial scale processing, such as for bauxite and spodumene minerals. However, these methods are based on using hydroxide solutions rather than molten hydroxides.

[00116] When 10 wt% of silica or sodium silicate powder was added to a molten hydroxide eutectic (1:1 NaOH:KOH) at 310 °C the silica or sodium silicate immediately started to react with the hydroxide, releasing water as a part of the reaction. The release and evaporation of water cooled the top of the molten hydroxide to below its melting temperature. This caused the formation of a solidified sponge atop of the molten hydroxide, stalling further reaction of silica due to poor thermal conductivity. To overcome

this issue, silica was slowly added to the molten hydroxide over 48 hours, resulting in a clear and transparent solution.

[00117] The reaction of quartz with molten hydroxide has the same overall chemistry as silica but is significantly slower and thus does not cause the same issues with formation of a spongy crust/matrix. Practically, this allows leaching of quartz to be easily integrated in a dissolution-electrowinning (of silica) circuit.

[00118] Example 12 - Nickel Sulphide Ore and Concentrate

[00119] A West Australian nickel sulphide ore sample (2.0% Ni, 14.1% Fe, 0.2% Cu, 6.3% S and 9.9% MgO) with particle size less than 3mm was added to an NaOH:KOH eutectic melt (1:1 by weight) at 250 °C (6 wt% ore, 94 wt% hydroxide). The ore instantly started to dissolve and was completely dissolved after 3 minutes, turning the clear molten hydroxide into an orange/brown solution.

[00120] The dissolved ore solution was used as an electrolyte in an electrowinning experiment where magnetic depositions were achieved with potentials as low as 1.4 V. The magnetic deposits were washed in water and then immersed in 5.5 M HCl where hydrogen evolution was detected, confirming the metallic nature.

[00121] Example 13 - Nickel Sulphide Ore and Concentrate

[00122] A sample of nickel sulphide concentrate from a West Australian nickel sulphide ore (13.6% Ni, 38.7% Fe, 1.1% Cu, 32.8% S and 3.5% MgO) with particle size less than 1mm was added to an NaOH:KOH eutectic melt (1:1 by weight) at 250°C (3 wt% concentrate, 97 wt% hydroxide). The concentrate dissolved quickly, within 2 minutes, in the molten hydroxide forming a deep red-brown solution. The dissolution occurred without any evolution of gases or vapour. The solution was left under a Teflon lid for 24 hours at 250 °C. After this period, no precipitation was observed, and the colour remained unchanged but with increased intensity.

[00123] The dissolved concentrate solution was then used for electrowinning experiments. It was observed that electrochemical current flow had already started at cell

voltages of 0.6 V and reached a low-current plateau at 0.9 V, before increasing rapidly again at 1.2 V. Cathode depositions at 1.0 V formed a thin coating with recognisable copper colour, indicating that copper could be selectively deposited from the solution, however the current densities were limited by the diffusion of low-concentration copper species in the solution.

[00124] Cathode depositions from the same solution, obtained at 1.6 V and 1.8V cell voltage were magnetic and produced hydrogen when exposed to diluted HCl solutions. Notably, at a cell voltage of 1.8 V, current densities above 200 mA/cm² were measured, which indicates very high diffusivity of the dissolved metal species in the molten hydroxide solution.

[00125] Copper, nickel and iron in the sulphide ores and concentrate are contained in sulphide structures and their electrodeposition from the molten hydroxide solution confirms that the sulphide bonding structures have been broken during the dissolution. It is therefore anticipated that other sulphide ores, for example, copper-iron sulphide ores such as chalcopyrite and their concentrates, will undergo a similar dissolution process in molten hydroxides.

[00126] It is worth noting that iron only exists in oxidation state +2 in sulphide ores, where in most commercial oxides all (hematite and goethite) or most (magnetite) iron is in oxidation state +3. From an (electro)reduction perspective this means that significantly less current/energy is needed for reduction of iron from sulphide structures than from oxides.

[00127] While this invention has been described in connection with specific embodiments thereof, it will be understood that it is capable of further modifications. This application is intended to cover any variations uses or adaptations of the invention following in general, the principles of the invention and including such departures from the present disclosure as come within known or customary practice within the art to which the invention pertains and as may be applied to the essential features hereinbefore set forth.

[00128] As the present invention may be embodied in several forms without departing from the spirit of the essential characteristics of the invention, it should be understood that the above described embodiments are not to limit the present invention unless otherwise specified, but rather should be construed broadly within the spirit and scope of the invention as defined in the appended claims. The described embodiments are to be considered in all respects as illustrative only and not restrictive.

[00129] Various modifications and equivalent arrangements are intended to be included within the spirit and scope of the invention and appended claims. Therefore, the specific embodiments are to be understood to be illustrative of the many ways in which the principles of the present invention may be practiced. In the following claims, means-plusfunction clauses are intended to cover structures as performing the defined function and not only structural equivalents, but also equivalent structures.

[00130] When a Markush group or other grouping is used herein, all individual members of the group and all combinations and sub-combinations possible of the group members are intended to be individually included in the disclosure. Every combination of components described or exemplified herein can be used to practice the invention, unless otherwise stated.

[00131] Whenever a range is given in the specification, for example, a temperature range, a time range, or a composition or concentration range, all intermediate ranges and subranges, as well as all individual values included in the ranges given are intended to be included in the disclosure. It will be understood that any subranges or individual values in a range or subrange that are included in the description herein can be excluded from the claims herein.

[00132] As used herein, "comprising" is synonymous with "including," "containing," or "characterized by," and is inclusive or open-ended and does not exclude additional, unrecited elements or method steps. As used herein, "consisting of' excludes any element, step, or ingredient not specified in the claim element. As used herein, "consisting essentially of' does not exclude materials or steps that do not materially affect the basic and novel characteristics of the claim. The broad term "comprising" is intended to encompass the narrower "consisting essentially of' and the even narrower "consisting of."

Thus, in any recitation herein of a phrase "comprising one or more claim element" (e.g., "comprising A), the phrase is intended to encompass the narrower, for example, "consisting essentially of A" and "consisting of A" Thus, the broader word "comprising" is intended to provide specific support in each use herein for either "consisting essentially of' or "consisting of." The invention illustratively described herein suitably may be practiced in the absence of any element or elements, limitation or limitations which is not specifically disclosed herein.

[00133] One of ordinary skill in the art will appreciate that materials and methods, other than those specifically exemplified can be employed in the practice of the invention without resort to undue experimentation. All art-known functional equivalents, of any such materials and methods are intended to be included in this invention. The terms and expressions which have been employed are used as terms of description and not of limitation, and there is no intention that in the use of such terms and expressions of excluding any equivalents of the features shown and described or portions thereof, but it is recognized that various modifications are possible within the scope of the invention claimed. Thus, it should be understood that although the present invention has been specifically disclosed by examples, preferred embodiments and optional features, modification and variation of the concepts herein disclosed may be resorted to by those skilled in the art, and that such modifications and variations are considered to be within the scope of this invention as defined by the appended claims.

[00134] Each reference cited herein is incorporated by reference herein in their entirety. Such references may provide sources of materials; alternative materials, details of methods, as well as additional uses of the invention.

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CLAIMS

- 1. A method of providing an ore concentrate solution suitable for beneficiation processing, the method including the step of (i) contacting an ore with one or more metal bases at elevated temperature.
- 2. A method according to claim 1 wherein the one or more metal bases are alkali metal bases, preferably chosen from lithium hydroxide, sodium hydroxide, potassium hydroxide, rubidium hydroxide or caesium hydroxide, or alkaline earth bases, preferably chosen from calcium hydroxide, barium hydroxide or strontium hydroxide.
- 3. A method according to claim 1 wherein the one or more metal bases at elevated temperature form a super-alkaline media that partially or fully dissolves the ore.
- 4. A method according to claim 1 wherein the one or more metal bases at elevated temperature form a super-alkaline media comprising 45 wt% to 100 wt% sodium hydroxide and/or potassium hydroxide.
- 5. A method according to claim 1 wherein the elevated temperature is 160 °C to 400 °C, preferably 200 °C to 350 °C, more preferably 250 °C to 350 °C.
- 6. A method according to claim 1 wherein the ore is chosen from the group comprising one or more of: iron ore, preferably haematite, magnetite or goethite; aluminium containing ores; gold ores; manganese containing ores; lead ores; cobalt containing ores; uranium containing ores; copper containing; nickel containing ores, preferably nickel sulphide ores; silver containing ores; tin containing ores; silica ores and quartz.
- 7. A method according to claim 1 which further includes the step of adding silicates, preferably quartz, feldspar, mica, amphibole, pyroxene, olivine or aluminium-silicate to the combination of the ore and the one or more metal bases at elevated temperature.

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- 8. A method according to claim 1 wherein the one or more metal bases at elevated temperature form a super-alkaline media, and the super-alkaline media is subjected to short term heating at high temperature to drive off water.
- 9. A method according to claim 1 wherein the ore concentrate is fed to a beneficiation process, preferably an extractive metallurgical process.
- 10. A method according to claim 1 wherein the ore concentrate is subjected to the further steps of:
 - (ii) extracting one or more components of the ore from the ore concentrate solution;

and optionally,

- (iii) feeding the extracted ore concentrate solution to an extractive metallurgical process, preferably electrometallurgy, to deposit metal; and optionally,
 - (iv) passing the extracted ore concentrate solution to further beneficiation processes.
- 11. A method according to claim 1 wherein the ore concentrate is subjected to the further steps of:
 - (ii) forming a solution comprising dissolved components from the ore concentrate;
- (iii) removing the components from the ore concentrate solution; and optionally
 - (iv) forwarding the purified solution from step (iii) to a downstream beneficiation process.
- 12. A concentrated ore produced according to the method of any one of claims 1 to 8.
- 13. A metal, recovered by the beneficiation step of any one of claims 9 to 11.

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FIGURE 1

Technology base

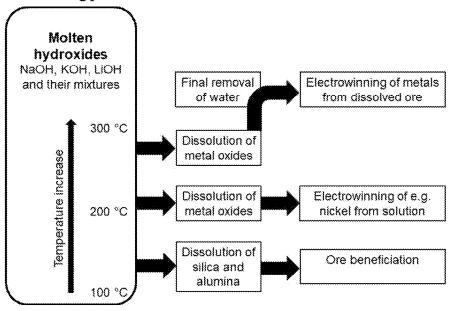
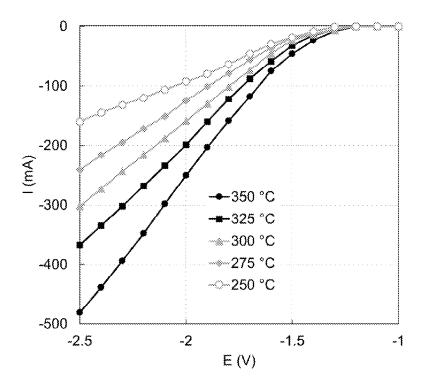
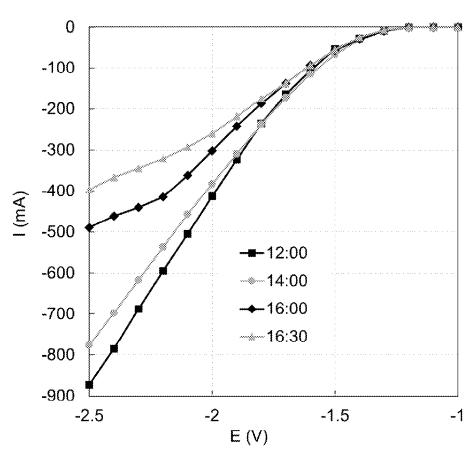


FIGURE 2



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FIGURE 3



INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU2023/051041

A. CLASSIFICATION OF SUBJECT MATTER

C22B 3/12 (2006.01) C01G 3/02 (2006.01) C01G 49/02 (2006.01) C01G 53/04 (2006.01) C22B 3/44 (2006.01) C25C 1/06 (2006.01) C25C 1/08 (2006.01)

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)

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)

Patenw: IPC/CPC: C01G49/06/low, C01G53/04/low, C01G53/11/low, C01G3/02/low, C01G3/12/low, C22B9/10/low, C22B3/44/low.

Espacenet, Google Patents & Google Scholar: Keywords (aqueous, molten, base, potassium hydroxide, sodium hydroxide, calcium hydroxide, rubidium hydroxide, caesium hydroxide, alkali, alkaline, metal hydroxide, iron ore, haematite, magnetite, goethite, aluminium ores, gold ore, manganese ore, lead ore, cobalt ore, uranium ore, copper core, nickel ore, nickel sulphide ore, silver ore, tin ore, silica ore, temperature, hot, heat, thermal, elevated, high & similar words).

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category* Citation of document, with indication, where appr		ropriate, of the relevant passages	Relevant to claim No.		
	Documents are listed in the continuation of Box C				
	X Fur	ther documents are listed in the continua	ation (of Box C X See patent family annotation	ex
considered to be of particular relevance "D" document cited by the applicant in the international application "E" earlier application or patent but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or "Y" document which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure use exhibition or other			later document published after the international filing date of in conflict with the application but cited to understand the punderlying the invention document of particular relevance; the claimed invention can novel or cannot be considered to involve an inventive step taken alone document of particular relevance; the claimed invention can involve an inventive step when the document is combined when the document is combine	not be considered when the document is not be considered to with one or more other	
Date of the actual completion of the international search		Date of mailing of the international search report			
6 December 2023		06 December 2023			
Name and mailing address of the ISA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA Email address: pct@ipaustralia.gov.au		Authorised officer Hatinder Sharma AUSTRALIAN PATENT OFFICE (ISO 9001 Quality Certified Service) Telephone No. +61 2 6225 6151			

	INTERNATIONAL SEARCH REPORT	Intern	national application No.
C (Continua	ion). DOCUMENTS CONSIDERED TO BE RELEVANT	PCT/	/AU2023/051041
Category*	Citation of document, with indication, where appropriate, of the relevant passages	I	Relevant to claim No.
X	US 2016/0194215 A1 (SUMITOMO METAL MINING CO., LTD) 07 July 2016 abstract; para 0005; para 0008; para 0015-0017; para 0025-0028; examples 1-3; figur	e I	1-13
WO 2013/010263 A1 (ORBITE ALUMINAE INC.) 24 January 2013 X abstract; para 0002; para 0009-0012; para 0029-0031; para 0045; para 0065-0072; para 0087; examples 1-2			1-13
US US 2016/0362304 A1 (SUMITOMO METAL MINING CO., LTD.) 15 December 2016 X abstract; para 0002; para 0050-0054; example 2; figures 1-2			1-3, 6, 8-13
A	US 2005/0072837 A1 (Nanis) 07 April 2005		1-13

Form PCT/ISA/210 (fifth sheet) (July 2019)

INTERNATIONAL SEARCH REPORT	International application No.
Information on patent family members	PCT/AU2023/051041

This Annex lists known patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document/s Cited in Search Report		Patent Family Member/s		
Publication Number	Publication Date	Publication Number	Publication Date	
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		US 9890055 B2	13 Feb 2018	
		AU 2014322310 A1	29 Oct 2015	
		AU 2014322310 B2	31 Mar 2016	
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		EP 3048083 B1	31 Jul 2019	
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		JP 2015059056 A	30 Mar 2015	
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		AU 2012286482 B2	02 Jul 2015	
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		CN 103958705 A	30 Jul 2014	
		CN 103958705 B	27 Jan 2016	
		CN 105543502 A	04 May 2016	
		EP 2734654 A1	28 May 2014	
		JP 2014523491 A	11 Sep 2014	
		JP 5909279 B2	26 Apr 2016	
		RU 2014105832 A	27 Aug 2015	
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US US 2016/0362304 A1	15 December 2016	None		
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		US 6953146 B2	11 Oct 2005	

Due to data integration issues this family listing may not include 10 digit Australian applications filed since May 2001. Form PCT/ISA/210 (Family Annex)(July 2019)

No. NSD527 of 2024

Federal Court of Australia

District Registry: New South Wales

Division: General

FORTESCUE LIMITED (ACN 002 594 872) and others

Applicants

ELEMENT ZERO PTY LIMITED (ACN 664 342 081) and others

Respondents

ANNEXURE COVER SHEET

This is **Annexure PAD-81** produced and shown to **PAUL ALEXANDER DEWAR** at the time of affirming his affidavit.

Before me:

ORE PROCESSING METHOD FOR METAL RECOVERY

FIELD OF INVENTION

[0001] The present invention relates to the field of ore processing

[0002] In one form, the invention relates to processing ores to provide valuable metal products.

[0003] In one particular aspect the present invention is suitable for recovery of metal from ores by chemical conversion, dissolution and electrodeposition.

[0004] It will be convenient to hereinafter describe the invention in relation to iron ore, particularly the reduction of iron from haematite ore however it should be appreciated that the present invention is not so limited but extends to a wide range of ores, and a wide range of metals.

BACKGROUND ART

[0005] It is to be appreciated that any discussion of documents, devices, acts or knowledge in this specification is included to explain the context of the present invention. Further, the discussion throughout this specification comes about due to the realisation of the inventor and/or the identification of certain related art problems by the inventor. Moreover, any discussion of material such as documents, devices, acts or knowledge in this specification is included to explain the context of the invention in terms of the inventor's knowledge and experience and, accordingly, any such discussion should not be taken as an admission that any of the material forms part of the prior art base or the common general knowledge in the relevant art in Australia, or elsewhere, on or before the priority date of the disclosure and claims herein.

[0006] Traditionally, reduction of metals such as iron from ore, particularly haematite ore, is done in a pyrometallurgical process which produces significant greenhouse gases, particularly carbon dioxide. Carbon is alloyed with iron to make steel as part of this process (normally to excessive levels when first reduced in a blast furnace) to produce pig iron and then reduced by oxidation in a basic oxygen steelmaking furnace. More recently, a direct

reduced iron (DRI) process has been progressively displacing blast furnace capacity. While this pathway is offers reduced carbon dioxide emissions, it is far from being carbon neutral. The DRI process uses high operating temperatures and has high inefficiencies. In addition, the process also does not work well with low grade ores (i.e., below 58 wt% Fe).

[0007] The alternative treatment methods typically include extractive metallurgy to remove metals from natural mineral deposits. Extractive metallurgy techniques are commonly grouped into three categories: hydrometallurgy, pyrometallurgy and electrometallurgy including electrorefining and electrowinning. Many of these processes also use high operating temperatures and have high inefficiencies.

SUMMARY OF INVENTION

[0008] An object of the present invention is to provide an improved process for extraction of metal from ore.

[0009] It is a further object of the embodiments described herein to overcome or alleviate at least one of the above noted drawbacks of related art systems or to at least provide a useful alternative to related art systems.

[0010] In a first aspect of embodiments described herein there is provided a method of separating metal from ore comprising the steps of:

- (a) optionally pre-processing the ore,
- (b) mixing the ore with a molten caustic medium to dissolve at least one metal species from the ore,

and

- (c) subjecting the molten mixture to electrolysis to deposit at least one metal on a cathode and evolve oxygen at an anode.
- [0011] The caustic medium may be circulated in the process, with a small bleed required to remove impurities.
- [0012] The molten caustic medium preferably comprises one or more alkali metal or alkaline earth bases. Alkali metal or alkaline earth bases suitable for use in the present invention are preferably hydroxides, although other bases such as metal oxides or metal

ammonium species may also be used. In one particularly preferred embodiment, the molten caustic medium is sodium hydroxide (NaOH).

[0013] The ore fed into the process of the present invention is typically subjected to pre-processing steps including crushing and drying to remove moisture as well as water bound in the lattice structure. In a second aspect of embodiments described herein there is provided a method for the separating metal from ore comprising the steps of:

- (a)(i) crushing the ore to a desired particle size;
- (a)(ii) drying the crushed ore;
- (b) mixing the crushed, dried ore with a caustic medium at elevated temperature, preferably molten caustic medium;
- (c) passing the mixture through electrolysis cells to collect metal on a cathode and release oxygen at an anode;
- (d) removing the metal from the cathodes or using cathode starter-sheets of the same material as the metal collected.

[0014] It will be apparent to the person skilled in the art that a very wide range of ores can be processed according to the present invention. Preferably, ore for use in the present invention is chosen from the group comprising one or more of the following: iron ore including hematite, goethite, magnetite, titanomagnetite and pisolitic ironstone; aluminium containing ores including bauxite, cryolite and corundum; gold ores including goldpolysulfide, gold-quartz, gold-telluride, gold-tetradymite, gold-antimony, gold-bismuthsulfosalt, gold-pyrrhotite, and gold-fahlore; manganese containing ores such as romanechite, manganite hausmannite and rhodochrosite; lead ores including galena, cerussite and anglesite; zinc ores including calamine and smithsonite; cobalt containing ores; uranium containing ores; copper containing ores including copper pyrite, malachite, cuprite and copper glance; nickel containing ores such as laterites and magmatic sulphide deposits; titanium containing ores; tungsten containing ores; silicon containing ores; rare earth containing ores; chromium containing ores; silver containing ores such as argentite; tin containing ores such as cassiterite, tinstone, stannite or cylindrite; heavy sands and quartz. In a particularly preferred embodiment the ore is iron ore, which is particularly rich

in iron oxides, particularly in the form of magnetite (Fe₃O₄), hematite (Fe₂O₃), goethite (FeO(OH)), limonite (FeO(OH)·n(H2O)) or siderite (FeCO₃).

[0015] The molten metal base or bases at elevated temperature may comprise a super-alkaline media. Upon contact with the metal base, metal containing moieties are chemically converted to solvable species and/or the resulting species are fully or partially dissolved in the molten metal base electrolyte. Without wishing to be bound by theory it is believed that sulphide ores, for example, are converted to oxides.

[0016] Additional compounds may facilitate the dissolution or chemical conversion of the ore. In particular, the addition of silicates may promote dissolution or chemical conversion of the ore.

[0017] The process of the present invention may include one or more bleed steams. Some steps of the process will build up materials such as solid fines, slimes, or breakdown products from chemicals. Their presence can lead to degradation of the overall performance of the process. Having a bleed from the relevant step removes these products and helps to maintains process performance.

[0018] Optimally, the bleed flow will be sufficient to prevent major build-up of the aforementioned material, yet not so large as to waste useful material.

[0019] The bleed from the electrolysis step may be further processed and recirculated to the process of the present invention. For example, the bleed may be further processed to remove impurities so that the caustic medium can be returned to the leach. Alternatively, after further processing the bleed can be fed back between the dissolution step and the electrolysis step.

[0020] Removal of impurities from the bleed may be carried out, for example, using a gravimetric approach to separate species having different densities.

[0021] In another aspect of embodiments described herein there is provided a method for separating metal from ore comprising the steps of:

- (a)(i)- crushing the ore to a desired particle size;
- (a)(ii)- drying the crushed ore;
- (b) mixing the crushed, dried ore with a caustic medium at elevated temperature, preferably molten;
- (c) passing the mixture through electrolysis cells to collect metal on a cathode and evolve oxygen at an anode, and spent caustic liquor;
- (d) removing the collected metal;
- (e) bleeding and cooling spent liquor from the electrolysis cells;
- (f) separating the spent liquor into a crud comprising impurities and cleaned caustic medium;

and

(g) returning the cleaned caustic medium to the mixing step.

[0022] The metal that collects on the cathode may be removed from the cathodes or starter sheets. Starter sheets are cathodes made of metals such as iron or steel, that are removed from the process and may be sold in combination with the deposited metal.

[0023] As the spent liquor cools, impurities coalesce and solidify in the upper level, leaving the relatively clean caustic medium in the lower levels. The process may include the further steps of;

- (h) crushing any solid crud,
- (i) milling the crud in liquid to form a suspension;
- (j) thickening the suspension of crud;

and

(k) drying, preferably spray drying, the thickened suspension.

[0024] Other aspects and preferred forms are disclosed in the specification and/or defined in the appended claims, forming a part of the description of the invention.

[0025] In essence, embodiments of the present invention stem from the realization that a caustic medium can be used to convert ore to a liquid that can be electrochemically treated to deposit valuable metals onto a cathode. More particularly, the realisation extends to the fact that no carbon need to be used in the process. When renewable energy is applied, no carbon dioxide is released to the atmosphere in any direct or supporting step.

[0026]	Advantages provided by the present invention comprise the following:
	can be carried out at ambient pressure;
	low electrowinning potential leading to low energy consumption;
	recycling of processing media and by-products leading to reduced waste;
	low emission;
	low operating temperature leading to the ability to easily follow intermittent
renew	able energy loads;
	low capital and operating costs;
	can be used to process a broad range of ores ranging from very low to very high
metal	content;
	includes a highly selective electrochemical process;
and	
	provides high purity products including high purity metal (>98 wt% purity).

[0027] Further scope of applicability of embodiments of the present invention will become apparent from the detailed description given hereinafter. However, it should be understood that the detailed description and specific examples, while indicating preferred embodiments of the invention, are given by way of illustration only, since various changes and modifications within the spirit and scope of the disclosure herein will become apparent to those skilled in the art from this detailed description.

BRIEF DESCRIPTION OF THE DRAWINGS

[0028] Further disclosure, objects, advantages, and aspects of preferred and other embodiments of the present application may be better understood by those skilled in the relevant art by reference to the following description of embodiments taken in conjunction

with the accompanying drawings, which are given by way of illustration only, and thus are not limitative of the disclosure herein, and in which:

FIG 1 illustrates the steps of the process of the current invention;

FIG 2 illustrates in more detail, suitable apparatus and plant configurations for use in the process of the present invention;

FIG 3 is a plot of iron ore solution concentration (wt%) against iron content in the dried iron ore; and

FIG 4 is a plot of current vs voltage for electrodeposition of iron from a hydroxide eutectic to illustrate energy consumption of the present invention.

[0029] <u>List of Parts</u>

	1 -		
1	Ore reclaim step	7	Bleed electrowinning step
11	Ore stockpile	71	Bleed electrowinning cell
12	Front end loader		
13	Feed bin		
14	Ore reclaim conveyor		
2	Grinding step	8	Bleed cooling step
21	Fine ore bin	81	Jacketed agitating tank
22	Belt feeder		
23	Cyclone		
3	Drying step	9	Crud crushing step
4	Chemical conversion step	10	Crud milling step
41	Screw feeder	101	Crud grinding mill
42	Feed leach box		
43	Leach tank 1		
44	Leach tank 2		
45	Leach tank 3		
46	Leach tank 4		
5	Electrowinning step	11	Spray drying step

51	Blank prep plant	111	Spray dryer
52	Electrowinning cells		
6	Product export step		

DETAILED DESCRIPTION

[0030] It is to be understood that the specific devices and processes illustrated in the attached drawings and described in the following specification are simply exemplary embodiments of the inventive concepts defined in the appended claims. Hence, specific dimensions and other physical characteristics relating to the embodiments disclosed herein are not to be considered as limiting, unless the claims expressly state otherwise. Additionally, unless otherwise specified, it is to be understood that discussion of a particular feature of component extending in or along a given direction or the like does not mean that the feature or component follows a straight line or axis in such a direction or that it only extends in such direction or on such a plane without other directional components or deviations, unless otherwise specified.

Process Overview

[0031] FIG 1 is a diagram illustrating the following process steps of the present invention:

ore reclaiming step (1) - Ore is reclaimed from an ore stockpile and conveyed or otherwise transported to a mill for dry crushing.
 ore grinding step (2) - The ore is ground in a mill.
 ore drying step (3) - Ground ore from the mill is subjected to a drying step, which may include for example, passing through a dry cyclone. Oversize material may be returned to the mill for regrinding until it reaches the desired target size.
 chemical conversion step (4) - The dry product is mixed into a caustic medium, such as a molten alkali metal or alkali earth at a volume flow rate that will allow proper

mixing. Apparatus such as a series of cascade mixing tanks may be used to ensure

a controlled chemical conversion followed by dissolution of solids.

9 electrowinning step (5) - The molten product is subjected to electrowinning. For example, the molten product may be passed through electrowinning cells where the metal product is collected on nickel or mild steel cathodes. The cathode material may be chosen to optimise efficiency of removal of the metal. □ isolation of final product (6) - The metal product is then removed from the cathodes and suitably packaged for transport by road or water. [0032] Optionally the spent caustic media is bled from the electrowinning step, the impurities are removed, and the remaining cleaned caustic material is returned for use or as a 'top up' reagent at a convenient point in the process such as the chemical conversion step or the dissolution step, or after the electrowinning step. Separation of the impurities as a crud from the caustic material is preferably carried out according to the following steps: □ bleed electrowinning step (7) - The spent caustic medium that is left when metal and oxygen have been removed during electrowinning may be bled and replaced with fresh caustic medium to avoid build-up of impurities. □ bleed cooling (8) - The bleed stream is cooled, and impurities form on the upper level of the cooling liquid. This may, for example, be carried out in "crud" baths. The relatively clean, solid, caustic forming the bottom layer is remelted and returned to the chemical conversion step with "top-up" reagent. □ crud crushing (9) - The upper layer of crud comprising impurities is crushed crud milling (10) - The crushed crud is then milled in water before the resultant stream is thickened. □ crud drying (11) - The thickened crud stream is dried, for example, by spray drying methods. The dried impurities may then be used for other purposes or other processes. For example, they may be used as feedstock for creating value-added products such as geopolymers or zeolites. They may alternatively be subjected to further processing such as electroreduction to recover other valuable metals (such as aluminium or silicon) or metalloids.

[0033] The steps of the present invention are now described in greater detail with reference to the embodiment of the process that is illustrated in FIG 2.

[0034] **Step 1 - Ore Reclaim**

[0035] The object of ore reclaim is to deliver ore fines at a controlled rate to a crusher. For example, fines may be delivered by trucks to a stockpile on a prepared pad situated close to the crushing facility and stockpiled adjacent a feed bin. Ore fines can be loaded into the feed bin by a front-end loader or by direct tipping or other suitable means.

[0036] Loading of fines to the feed bin can be controlled by any convenient means, such as a crusher operator who activates tipping from a suitable location such as a central control room. Typically, a feeder, such as an apron feeder under the feed bin discharges to a sacrificial conveyer which then discharges to an enclosed belt conveyer.

[0037] The primary crusher discharge conveyor may include a tramp metal detection and a belt magnet to remove extraneous metal from potentially damaging downstream equipment.

[0038] Preferably, dust emission control in the ore reclaim area is provided by an extraction system within the reclaim structure. The dust emission control system may for example, consist of a single dry bag house and the dust can be discharged back to the enclosed conveyor belt. A sump pump may be provided in the basement level of the fines reclaim building to remove dust collected by washdown of the primary crushing area.

[0039] **Step 2 - Ore Grinding**

[0040] In a preferred embodiment belt feeders are installed beneath the fine ore bin and discharge to respective high pressure grinding roll (HPGR) crushers. A variable speed drive in each feeder may be used to maintain the level in the HPGR feed chute which ensures that the HPGR remains choke fed in normal operation

[0041] Tramp metal, if detected by a dedicated metal detector on each feeder, may actuate a flop gate in the HPGR belt feeder discharge chute. A metal detection can lead to diversion of the individual feeder discharge to a HPGR bypass conveyor. The stream containing the metal can then pass under a bypass magnet that removes the metal and the conveyor discharge can gravitate back to the HPGR discharge conveyor. This system is designed to allow the crushers to remain online in the event of a metal detection event.

[0042] An HPGR typically has two rolls driven by synchronised variable speed drives which control the HPGR crusher throughput. The crusher gap is typically controlled by hydraulic pressure on the movable rolls. Tertiary crushed ore can gravitate directly to an HPGR discharge conveyor which conveys the crushed product to a two-stage air classification circuit.

[0043] Preferably, oversize particles are removed from the ground ore. For example, ground ore from mill may be passed through a dry cyclone and oversize particle can be returned to mill for regrinding to target size.

[0044] **Step 3 - Ore Drying**

[0045] The crushed ore may be passed through a dryer to remove water of crystallisation before the ore is fed to the chemical conversion step. The leach feed is typically dried at elevated temperature and the off gas from the dryer can be used in part in the crushing circuit for heating and product transport, and in part in the spray dryer to evaporate water from the caustic waste solutions.

[0046] The dry product is collected and conveyed in part to the chemical conversion step and in part to the bleed

[0047] Step 4 - Chemical conversion

[0048] In a preferred embodiment, dried and crushed iron ore may be fed from the dryer to a leach feed box by screw feeders which mix all feed streams to the fines leach circuit through operation of dart valves. The dry product may be mixed into to molten caustic medium at elevated temperature and at a volume flow rate that will allow proper mixing.

[0049] Chemical conversion may be carried out by many means. In a preferred embodiment a series of cascade mixing tanks ensures a controlled dilution of solids. Agitated atmospheric leach tanks, with leach agitators may provide the necessary mixing and solids suspension for optimum reaction kinetics. The last tank in the train may discharge to a pump box from where the leach discharge is pumped by the duty leach discharge pump to an electrowinning circuit.

[0050] Typically, the caustic medium is a metal base chosen from alkali metal bases such as lithium, sodium, potassium, rubidium or caesium hydroxide, or alkaline earth bases such as calcium, barium, or strontium metal hydroxides. In a particularly preferred embodiment, the metal base is chosen from lithium hydroxide, sodium hydroxide, potassium hydroxide or calcium hydroxide. In a particularly preferred embodiment, the caustic medium comprises 45 wt% to 100 wt% sodium hydroxide and/or potassium hydroxide.

[0051] One or more metal bases may be contacted with the ore, and combinations of metal bases may be in the form of a eutectic mixture. Eutectic mixtures of sodium, potassium and/or lithium hydroxide is particularly preferred. In some embodiments, for economic reasons NaOH is preferred but pure NaOH may not be as efficient as the combination of NaOH with KOH to form a eutectic system, which allows for lower operating temperature.

[0052] The caustic media comprising alkali metal or alkaline earth bases are contacted with the ore at elevated temperature, preferably a temperature above 160 °C, or above 200 °C, preferably above 250 °C, more preferably above 300 °C. In a particularly preferred embodiment, the alkali metal or alkaline earth bases are contacted with the ore at a temperature of 160 °C to 400 °C, preferably 200 °C to 350 °C, more preferably 250 °C to 350 °C.

[0053] For example, with reference to eutectics, most mixtures of NaOH and KOH have lower melting points than the constituent compounds. For a 1:1 molar ratio of NaOH:KOH, the eutectic forms at 170 °C. If adsorbed water is present, such as in a 1:1:1 ratio of NaOH:KOH:H₂O, the temperature of formation of the eutectic can be below 100 °C.

[0054] Molten metal bases, particularly hydroxides often include impurities such as water. Preferably metal bases incorporated in the super-alkaline media of the present invention will include water in amounts of no more than one mole of water per more of hydroxide. It is also possible to drive off water from the super-alkaline media by short term heating of the super-alkaline media to higher temperatures (i.e., > 500 °C). A shield of inert gas over the super-alkaline media can then be used to restrict or prevent reabsorption of water.

[0055] The metal bases used in the present invention may include small amounts of chemical impurities. For example, sodium hydroxide may form, or include small amounts of sodium carbonate (Na₂(CO₃)).

[0056] Step 5 - Electrowinning

[0057] Preferably, the metal is recovered by passing the molten product through electrowinning cells where the metal is collected on a cathode. In the electrowinning cell or cells, the anode is preferably positioned higher than cathode. Separation efficiency of deposited solid-state iron product from cathode will determine the best cathode material to use.

[0058] Preferably, multiple identical tank houses are used to strip metal from the molten caustic slurry. Each tank house may comprise multiple banks of electrowinning cells. The molten slurry within each cell can be distributed via an individual manifold located at the base of each cell.

[0059] As the metal is electroplated, sodium and potassium hydroxide are regenerated however several side reactions may consume additional regent.

[0060] Plating takes place over a cycle time that depends on the desired thickness of metal on the cathode.

[0061] The unavoidable evolution of oxygen at the anodes would tend to give rise to an effervescent bubbling effect at the electrolyte surface. This potentially causes a "caustic mist" which is preferably removed by a ventilation system, which covers the cells to prevent lead aerosols in the immediate area.

[0062] A critical operation impacting on the current efficiency of an electrowinning cell house is that of detecting and correcting short circuits ("shorts") between anode / cathode pairs and poor contacts. Shorts and poor contacts can be individually identified, allowing the operator to take remedial action. Alternatively, hand-held gauss meters can be employed.

[0063] Step 6 - Final Product Removal

[0064] Cathode handling typically involves lifting a proportion of the plated cathodes from each cell at a time, such as by using an overhead cathode stripping crane and lifting cradle and then transporting them to cathode storage conveyers.

[0065] In one embodiment of the present invention, the metal deposit is removed from the cathode so that the cathodes can optionally be re used.

[0066] Alternatively, the cathodes may be sacrificial and remain combined with the deposited metal. The cathodes can be cleaned of molten caustic media with hot water sprays and the washed cathodes stacked. The hanger bars may be automatically stripped from the cathodes and passed to the starter sheet package bin for reuse. Starter sheets that have previously been prepared can be used to replace the cathodes removed.

[0067] The cathodes can then be weighed and bundled for shipment.

[0068] Step 7 - Bleed Electrowinning

[0069] A bleed stream may be taken from the main electrowinning circulating flow and is subjected to electrowinning to remove most of the leached iron in this stream. As the metal is plated, sodium and potassium hydroxide are regenerated in the bleed flow.

[0070] Most of the water generated in electrowinning would be consumed in regenerating sodium and potassium hydroxide. Optimally, the bleed electrolyte is stripped to the minimum level possible consistent with the number of electrowinning cells used and current efficiency.

[0071] Water balance in the process is principally affected by three different steps which are explained as follows, using iron ore as the exemplary ore. When iron ore is processed according to the present invention there are three main products (silica, Fe(II) and Fe(III)) in the dissolution process which have three different chemical "routes" that affect water balance:

1) The silica "route" releases water all the way through the process according to the following equations:

Dissolution: $2Fe_2O_3 + 4Na_2SiO_3 + 4H_2O \rightarrow 4FeSiO_3 + 8NaOH + O_2$

 $4SiO_2 + 8NaOH \rightarrow 4Na_2SiO_3 + 4H_2O$

Overall: $2Fe_2O_3 + 4SiO_2 \rightarrow 4FeSiO_3 + O_2$

Deposition: $4\text{FeSiO}_3 + 8\text{NaOH} \rightarrow 4\text{Fe} + 4\text{Na}_2\text{SiO}_3 + 4\text{H}_2\text{O} + 2\text{O}_2$

2) The iron(II) "route" which is water neutral overall:

Dissolution: $2Fe_2O_3 + 4NaOH \rightarrow 4NaHFeO_2 + O_2$

Deposition: $4NaHFeO_2 \rightarrow 4Fe + 4NaOH + 2O_2$

Overall: $2Fe_2O_3 \rightarrow 4Fe + 3O_2$

3) The iron(III) "route" which requires water during dissolution, but which is water neutral overall:

Dissolution: $2Fe_2O_3 + 4NaOH + 2H_2O \rightarrow 4NaH_2FeO_3$

Deposition: $4NaH_2FeO_3 \rightarrow 4Fe + 4NaOH + 2H_2O + 3O_2$

Overall: $2Fe_2O_3 \rightarrow 4Fe + 3O_2$

Bleed Treatment

[0072] Step 8 - Bleed Cooling

The bleed electrowinning discharge can be treated with ground ore to react the sodium and/or potassium silicates (which are water soluble) to iron silicate which is not water soluble. Sodium and potassium oxides can also react which further improves sodium and potassium hydroxide recovery. Reaction of residual hydroxide with ore is one possible way to neutralise the 'waste' stream.

[0073] The slurry is then cooled, such as in a jacketed agitated tank in closed circuit with a cooling tower. Cooling a molten slurry causes the multiple phases in solution in the molten caustic to separate out. Once separation is complete molten caustic medium is pumped from the bottom of the kettle. Molten crud is pumped from the top of the kettle and allowed to solidify.

Crud Treatment

[0074] Step 9 - Crud Crushing

[0075] The crud produced by cooling of the electrowinning bleed stream can be recovered, stockpiled, and loaded into a crud crushing feed bin. Loading of crud into the feed bin may be controlled by the crusher operator from the central control room.

[0076] An apron feeder or similar under the feed bin may discharge to the crud crusher to produce a product suitable to feed the semi-autogenous grinding (SAG) mill.

[0077] **Step 10 - Crud Milling**

[0078] The solidified crud can be milled in a mill to allow the sodium and potassium oxides in the crud to be dissolved for recovery. The mill discharge can be cycloned to impart particle size classification.

[0079] Cyclone underflow can be recycled to a mill feed chute. Cyclone overflow will gravitate to a crud thickener where the undissolved solids can be recovered to the thickener underflow. Thickener overflow is pumped to a spray dryer.

[0080] Thickener underflow can be pumped to a filter surge tank to provide a buffer between the milling and filtration circuits. The thickener underflow can be filtered and washed with wash liquor and filtrate is recycled to the crud thickener. Washed filter cake should be suitable for disposal.

[0081] **Step 11 - Spray Drying**

[0082] The combined liquor streams from the crud milling and cathode washing can be treated in a spray dryer to recover the sodium and potassium hydroxide as a solid product for recycle to the leach. Waste heat from the ore dryer may be used in the spray dryer to evaporate the water.

[0083] **EXAMPLES**

[0084] The present invention will be further described with reference to the following non-limiting examples.

[0085] **EXAMPLE 1 - Processing of Iron Ore**

[0086] The present invention will be further explained with reference to the following example in which iron was recovered from Marra Mamba style fine ores (-6 mm) that are found in the Pilbara, Western Australia, using a 5 Mtpa modular process plant. Marra Mamba ore has about 58% iron content and less than 1% moisture when particle size is less than 6 mm. Impurities include silica, alumina, and magnesia as well as trace content of phosphorus and titania.

[0087] **Step 1 - Ore reclaim**

[0088] Marra Mamba type -6 mm iron fine ore stockpile was conveyed by trucks to a mill and stockpiled. Front end loaders loaded fines to a feed bin. An ore reclaim conveyor discharged into an 1850 tonne live capacity fine ore bin providing approximately one (1) hour live capacity ahead of the crushing circuit. Withdrawal rate from the fine ore bin was matched to the crushing circuit availability of 87.4% corresponding to an average feed rate of 653 dry metric tonnes per hour in normal operation. Live capacity of the feed bin was about 650 tonnes giving approximately 1 hour surge capacity at design reclaim throughput.

[0089] Belt feed conveyer belts were installed beneath the fine ore bin for discharge to respective high pressure grinding roll (HPGR) (1200 CRR-0001, 0002) crushers. A variable speed drive on each feeder was used to maintain the level in the HPGR feed chute to ensure that the HPGR remained choke fed in normal operation.

[0090] **Step 2 - Ore grinding**

[0091] The -6 mm iron or was crushed to 1 mm average particle size. Ground ore from crusher was passed through a dry cyclone and +1 mm oversize was returned to crusher for regrinding to target size.

[0092] **Step 3 - Ore drying**

[0093] Marra Mamba ore typically has less than about 1% moisture. The crushed ore was passed through a dryer to remove water of crystallisation before the ore was fed to the chemical conversion step. The leach feed was dried at a temperature of 700 °C.

[0094] Step 4 - Chemical Conversion

[0095] The dry product was mixed at <18% solids to melted NaOH (325 °C to 350 °C) at a volume flow rate that allowed proper mixing. The consumption rate of sodium

hydroxide was approximately 90 kg/tonne of ore. The caustic NaOH media was required to be heated to processing temperature prior to adding ore, because the mixture tends to solidify quickly. The proportion of solids added into the mixture had a maximum of 18 wt% at 58% iron ore grade. This maximum would be reduced if lower grade feed was used. The output concentration of the processing circuit was approximately 5 wt% dissolved ore.

[0096] A series of four cascade mixing tanks ensured a controlled dilution of solids down to approximately 5% solids. Nickel lined vessels were appropriate for this chemistry and temperature. It will be readily apparent to the person skilled in the art that the vessels can be made of any convenient material, such as nickel 200, stainless steel with metal lining, Hastelloy® such as Hastelloy® C-276 or single crystal corundum. Other materials such as zinc oxide, cerium(IV) oxide and nickel oxide may be suitable materials for manufacture of vessels, but their suitability depends on their mechanical properties. Mixing efficiency was important along with maintaining heat in the leach tanks and leach agitators provided the necessary mixing. The last tank in the cascade discharged to a pump box from where the leach discharge was pumped by the duty leach discharge pump to a distribution circuit which split the leach discharge equally between five electrowinning circuits.

[0097] Without wishing to be bound by theory, it is believed that the chemistry occurring during the electrowinning step comprised the following major reactions:

$$2Fe_2O_3 + 4NaOH \rightarrow 4NaHFeO_2 + O_2$$

 $2Fe_2O_3 + 4KOH \rightarrow 4KHFeO_2 + O_2$
 $Fe_2O_3 + 4NaOH \rightarrow 2NaH_2FeO_3 + Na_2O$
 $Fe_2O_3 + 4KOH \rightarrow 2KH_2FeO_3 + K_2O$
 $2Fe_2O_3 + 4Na_2SiO_3 \rightarrow 4FeSiO_3 + 4Na_2O + O_2$
 $2Fe_2O_3 + 4K_2SiO_3 \rightarrow 4FeSiO_3 + 4K_2O + O_2$
 $2Fe_2O_3 + 4K_2SiO_3 \rightarrow 4FeSiO_3 + 4K_2O + O_2$
 $2Fe_2O_3 + 4K_2SiO_3 \rightarrow 4FeSiO_3 + 4K_2O + O_2$
 $2Fe_2O_3 + 2NaOH \rightarrow Na_2SiO_3 + H_2O$
 $2Fe_2O_3 + 2KOH \rightarrow 2NaAIO_2 + H_2O$
 $2Fe_2O_3 + 2NaOH \rightarrow 2NaAIO_2 + H_2O$
 $2Fe_2O_3 + 2KOH \rightarrow 2KAIO_2 + H_2O$

 $Na_2O + H_2O \rightarrow 2NaOH$ $K_2O + H_2O \rightarrow 2KOH$

[0098] Step 5 - Electrowinning

[0099] Recovery of deposited solid-state iron was via electrowinning onto a cathode. The molten product was passed through electrowinning cells where the product is collected on nickel or mild steel cathodes.

[00100] Five (5) identical tank houses were provided to strip iron from molten caustic slurry. Each tank house comprised four banks of electrowinning cells of 69 cathodes and 70 anodes each. It will be readily apparent to the person skilled in the art that the electrowinning tanks can be made of any convenient material, such as nickel 200, stainless steel with metal lining, Hastelloy® such as Hastelloy® C-276 or single crystal corundum. Other materials such as zinc oxide, cerium(IV) oxide and nickel oxide may be suitable materials for manufacture of vessels, but their suitability depends on their mechanical properties. Each cathode had 2 m² of plating area. The overall electro-winning cell houses were designed for an iron cathode production of 2,870,000 tpa, plus the additional iron electrowon in the bleed electrowinning circuit used for the impurity removal circuit. Each bank had a dedicated rectifier operating at a voltage of 204 V and a current of 276,000 A. The molten slurry within each cell was distributed via an individual manifold located at the base of each cell at a flow rate of between 1.80 l/min/m² based on a current density of 1000 A/m².

[00101] As the metal was plated, sodium and potassium hydroxide were regenerated however several side reactions consumed additional regent.

[00102] Solution from the cell overflows into a common pipe header by which the molten slurry gravitates to a heated circulation tank.

[00103] Plating took place over a cycle time that was dictated by the desired cathode thickness of approximately 12 mm, which resulted in a maximum cathode weight of 425 kg.

[00104] Without wishing to be bound by theory it is believed that the relevant chemistry comprises the following major reactions:

$$2NaHFeO_2 \rightarrow 2Fe + 2NaOH + O_2$$

 $2KHFeO_2 \rightarrow 2Fe + 2KOH + O_2$
 $4NaH_2FeO_3 \rightarrow 4Fe + 4NaOH + 2H_2O + 3O_2$
 $4KH_2FeO_3 \rightarrow 4Fe + 4KOH + 2H_2O + 3O_2$
 $2FeSiO_3 + 4NaOH \rightarrow 2Fe + 2Na_2SiO_3 + 2H_2O + O_2$
 $2FeSiO_3 + 4KOH \rightarrow 2Fe + 2K_2SiO_3 + 2H_2O + O_2$

[00105] Step 6 - Final Product Removal

[00106] A thin sheet of steel was guillotined to produce starter sheets of suitable size for cathodes. A handing bar was automatically fitted to the starter sheet before the prepared cathodes were stacked on a rack for use in the five tank houses. As electrowinning cells were stripped of loaded cathodes, a set of starter sheets is replaced in individual electrowinning cells as required.

[00107] Cathode handling involved lifting one third (15) of the plated cathodes from each cell at a time with an overhead cathode stripping crane and lifting cradle and then transporting them to a cathode storage conveyer. There they were cleaned of molten caustic with hot water sprays and the washed cathodes were transferred to conveyors feeding a stacking machine. The hanger bars were stripped from the cathodes and passed to the starter sheet package bin for reuse.

[00108] The cathodes of 'green' iron (>98%) are then ready for storage, shipment or further processing to produce various steel grades.

[00109] Bleed Treatment

[00110] The molten caustic electrowinning/chemical conversion media was bled to remove build-up of impurities. The bleed stream was cooled in "crud" baths. The impurities formed on the upper level of the "crud" baths. The relatively clean, now solid, caustic at the bottom was remelted and returned to the circuit with "top-up" (replenishing) reagent and the other caustic recovered in the aqueous circuit. The upper layer of crud was

crushed and then milled in water before the resultant stream was thickened and sent for disposal. The thickener overflow was sent to a spray dryer to recover itinerant caustic soda, with the resultant solids being returned to the process.

[00111] Step 7 - Bleed Electrowinning

[00112] A bleed stream was taken from the main electrowinning circulating flow and subjected to electrostripping to remove most of the leached iron in the stream. As the metal is plated, sodium and potassium hydroxide are regenerated in the bleed flow.

[00113] Without wishing to be bound by theory it is believed that the relevant reactions are as follows:

$$2NaHFeO_2 \rightarrow 2Fe + 2NaOH + O_2$$

$$2KHFeO_2 \rightarrow 2Fe + 2KOH + O_2$$

$$4NaH_2FeO_3 \rightarrow 4Fe + 4NaOH + 2H_2O + 3O_2$$

$$4KH_2FeO_3 \rightarrow 4Fe + 4KOH + 2H_2O + 3O_2$$

$$2FeSiO_3 + 4NaOH \rightarrow 2Fe + 2Na_2SiO_3 + 2H_2O + O_2$$

$$Na_2O + H_2O \rightarrow 2NaOH$$

$$K_2O + H_2O \rightarrow 2KOH$$

[00114] Step 8 - Bleed Cooling

[00115] The bleed electrowinning discharge is treated with ground ore to react the sodium and potassium silicates which are water soluble to iron silicate which is not water soluble. Sodium and potassium oxides are also reacted which further improves sodium and potassium hydroxide recovery.

[00116] Without wishing to be bound by theory it is believed that the relevant reactions are as follows:

$$2Fe_2O_3 + 4Na_2SiO_3 \rightarrow 4FeSiO_3 + 4Na_2O + O_2$$

 $2Fe_2O_3 + 4K_2SiO_3 \rightarrow 4FeSiO_3 + 4K_2O + O_2$
 $H_2O + Na_2O \rightarrow 2NaOH$

 $H_2O + K_2O \rightarrow 2KOH$

[00117] The slurry was then cooled to about 300 °C in a jacketed agitated tank in closed circuit with a cooling tower. Cooling the molten slurry to 300 °C caused the multiple phases in solution in the molten caustic to separate out. Several cooling kettles were provided to allow the separation to occur batchwise. Once separation was complete molten sodium and potassium hydroxide was pumped from the bottom of the kettle. Molten crud was pumped from the top of the kettle and allowed to solidify.

[00118] Step 9 - Crud Crushing

[00119] The crud produced by cooling of the electrowinning bleed stream was recovered and stockpiled. Lump crud was loaded into the crud crushing feed bin by a front-end loader, under the control of a crusher operator from a central control room. Live capacity of the crud crusher feed bin was 100 tonnes giving approximately 1 hour surge capacity at design reclaim throughput.

[00120] An apron feeder under the feed bin discharges to the crud crusher to produce a product suitable to feed a semi-autogenous grinding (SAG) mill.

[00121] **Step 10 - Crud Milling**

[00122] The solidified crud was milled in a single stage SAG mill to allow the sodium and potassium oxides in the crud to be dissolved for recovery. The SAG mill discharge was cycloned to impart classification.

[00123] Cyclone underflow was recycled to the SAG mill feed chute. Cyclone overflow gravitated to the crud thickener where the undissolved solids were recovered to the thickener underflow. Thickener overflow was pumped to a spray dryer circuit.

[00124] Thickener underflow was pumped to a filter surge tank to provide a buffer between the milling and filtration circuits. The thickener underflow was filtered and washed on two belt filters. Wash liquor and filtrate was recycled to the crud thickener. The washed filter cake was suitable for disposal.

[00125] **Step 11 - Spray dryer**

[00126] The combined liquor streams from the crud milling and cathode washing were treated in a spray dryer to recover the sodium and potassium hydroxide as a solid product for recycle to the leach.

[00127] **EXAMPLE 2 - Bench Scale Optimisation of Chemical Conversion of Iron**

[00128] Initial test work was carried out to optimise the dissolution of the iron ore used in Example 1. Test results suggested that the dissolution of ore occurs within a few minutes for dried (200 °C) ore with a particle size of 130 microns.

[00129] The maximum concentration of ore that could be dissolved in the molten caustic (the saturation point) has been measured at 300 °C for different ore qualities after one-and four-hours' exposure to the hydroxide. There was no appreciable difference in the amount of ore dissolved after one and four hours. The saturation point is dependent on the ore quality.

[00130] The more impure ore (with higher amount of silica and alumina) has higher saturation point. This is illustrated by the plot of FIG 3 which shows that the maximum concentration of ore in solution (i.e., saturation) increases with decreasing ore purity.

[00131] Control experiments were performed using pure silica and alumina dissolved in molten hydroxide. The saturation points for both were above 30% wt. This indicates that all silica and alumina in the ore samples is dissolved in molten hydroxide. However, the increase in saturation point with silica and alumina rich ore is significantly higher than the additional silica and alumina, so it appears that there is a synergistic effect from additional silica and alumina which is presumed to be linked to the formation of iron silicates and iron aluminates.

[00132] Other minerals present such as iron oxides or aluminium oxides may also react with NaOH to form additional salts in solution.

[00133] **EXAMPLE 3 - Bench Scale Electrowinning Optimisation**

[00134] Initial bench scale electrowinning test work was based on a cathode area of 8 cm² operating at 310 °C. A caustic medium of NaOH and KOH (5:1 mix ratio) was used to lower the melting point of the hydroxide.

[00135] It is anticipated that using NaOH as the sole component of the caustic medium is feasible and may offer advantages. In this case, the operating temperature would need to be increased to \sim 350 °C.

[00136] The test work found that ore loading in the 2 to 6 wt% range is provided a sufficiently low viscosity and at the same time provided enough iron in solution to maintain a current density in the 500 to 1000 A/m² range with cell voltages ranging from 1.6 V to 1.9 V. The spacing between cathode and anode was 2.5 cm.

[00137] FIG 4 is a plot of current vs voltage for electrodeposition of iron from hydroxide eutectic to illustrate energy consumption of the present invention. The eutectic was at 310 °C with 7 wt% dried Pilbara ore (55 wt% Fe) in solution.

[00138] The plot illustrates the energy consumption per ton of iron produced as a function of cell potential (upper trace) including 90% estimated Faradaic (current) efficiency. The lower trace represents the current density.

[00139] The coating deposited under these conditions on nickel or iron cathodes had an iron content between 95% and 98% depending on the ore quality. In these tests the dissolved silica and alumina was not removed from the molten hydroxide prior to the electrowinning and the applied voltage/current, where lower voltage/current always gave higher iron content. However, the impurity in the deposited iron was oxygen, not silica (or silicon) or alumina (or aluminium).

[00140] While this invention has been described in connection with specific embodiments thereof, it will be understood that it is capable of further modification(s). This application is intended to cover any variations uses or adaptations of the invention following in general, the principles of the invention and including such departures from the present disclosure as come within known or customary practice within the art to which the invention pertains and as may be applied to the essential features hereinbefore set forth.

[00141] As the present invention may be embodied in several forms without departing from the spirit of the essential characteristics of the invention, it should be understood that the above described embodiments are not to limit the present invention unless otherwise specified, but rather should be construed broadly within the spirit and scope of the invention as defined in the appended claims. The described embodiments are to be considered in all respects as illustrative only and not restrictive.

[00142] Various modifications and equivalent arrangements are intended to be included within the spirit and scope of the invention and appended claims. Therefore, the specific embodiments are to be understood to be illustrative of the many ways in which the principles of the present invention may be practiced. In the following claims, means-plusfunction clauses are intended to cover structures as performing the defined function and not only structural equivalents, but also equivalent structures.

[00143] "Comprises/comprising" and "includes/including" when used in this specification is taken to specify the presence of stated features, integers, steps or components but does not preclude the presence or addition of one or more other features, integers, steps, components or groups thereof. Thus, unless the context clearly requires otherwise, throughout the description and the claims, the words 'comprise', 'comprising', 'includes', 'including' and the like are to be construed in an inclusive sense as opposed to an exclusive or exhaustive sense; that is to say, in the sense of "including, but not limited to".

[00144] When a Markush group or other grouping is used herein, all individual members of the group and all combinations and sub-combinations possible of the group members are intended to be individually included in the disclosure. Every combination of components described or exemplified herein can be used to practice the invention, unless otherwise stated.

[00145] Whenever a range is given in the specification, for example, a temperature range, a time range, or a composition or concentration range, all intermediate ranges, and subranges, as well as all individual values included in the ranges given are intended to be included in the disclosure. It will be understood that any subranges or individual values in

a range or subrange that are included in the description herein can be excluded from the claims herein.

[00146] One of ordinary skill in the art will appreciate that materials and methods, other than those specifically exemplified can be employed in the practice of the invention without resort to undue experimentation. All art-known functional equivalents, of any such materials and methods are intended to be included in this invention. The terms and expressions which have been employed are used as terms of description and not of limitation, and there is no intention that in the use of such terms and expressions of excluding any equivalents of the features shown and described or portions thereof, but it is recognized that various modifications are possible within the scope of the invention claimed. Thus, it should be understood that although the present invention has been specifically disclosed by examples, preferred embodiments and optional features, modification and variation of the concepts herein disclosed may be resorted to by those skilled in the art, and that such modifications and variations are considered to be within the scope of this invention as defined by the appended claims.

[00147] Each references cited herein is incorporated by reference herein in their entirety. Such references may provide sources of materials, alternative materials, details of methods, as well as additional uses of the invention.

CLAIMS

- 1. A method of separating metal from ore comprising the steps of:
 - (a) optionally pre-processing the ore,
 - (b) mixing the ore with a molten caustic medium to dissolve at least one metal species from the ore,

and

- (c) subjecting the molten mixture to electrolysis to deposit at least one metal at a cathode and evolve oxygen at an anode.
- 2. A method for the separating metal from ore comprising the steps of:
 - (a)(i) crushing the ore to a desired particle size;
 - (a)(ii) drying the crushed ore;
 - (b) mixing the crushed, dried ore with a caustic medium at elevated temperature,preferably molten caustic medium;
 - (c) passing the mixture through electrolysis cells to collect metal at a cathode and release oxygen at an anode;
 - (d) removing the collected metal.
- 3. A method for the separating metal from ore comprising the steps of:
 - (a)(i)- crushing the ore to a desired particle size;
 - (a)(ii)- drying the crushed ore;
 - (b) mixing the crushed, dried ore with a caustic medium at elevated temperature, preferably molten;
 - (c) passing the mixture through electrolysis cells to collect metal on a cathode and evolve oxygen at an anode, and spent caustic liquor;
 - (d) removing the collected metal;
 - (e) bleeding and cooling spent liquor from the electrolysis cells;
 - (f) separating the spent liquor into a solid crud comprising impurities and cleaned caustic medium:

and

- (g) returning the cleaned caustic medium to the mixing step.
- 4. A process according to claim 3 which further includes the steps of;

- (h) crushing any solid crud,
- (i) milling the crud in liquid to form a suspension;
- (j) thickening the suspension;

and

(k) drying, preferably spray drying, the thickened suspension.

FIG 1

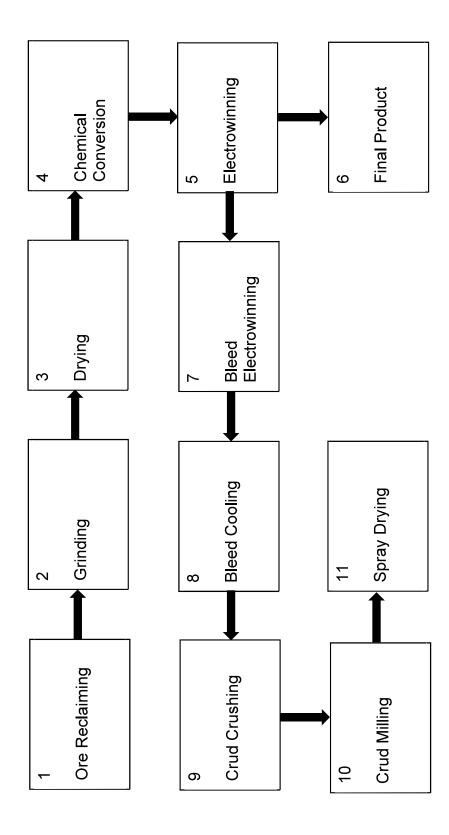


FIG 2

