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N.B. Use International abbreviation for country (See Schedule 4)	ZA 31	94/3729	32	Oth June, 1994
TITLE OF INVENTION				
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## DEPARTMENT OF TRADE AND INDUSTRY DEPARTEMENT VAN HANDEL EN NYWERHEID

REPUBLIC OF SOUTH AFRICA



REPUBLIEK VAN SUID-AFRIKA

## LETTERS PATENT

(PATENTS ACT, 1978)

### **PATENTBRIEF**

(WET OP PATENTE, 1978)

No. 9	5/4458		
1			
MUEDEAG		;	
WHEREAS NADEMAAL	MINTEK.		
		(Hereinafter called "the Patentee")	•

has applied to me for the grant of a patent in respect of an invention described and claimed in the complete specification aansoek by my gedoen het om die verlening van 'n patent ten opsigte van 'n uitvinding wat beskryf is en waarop aanspraak

deposited at the Patent Office under the above-mentioned number, a copy of which is annexed, together with the relevant gemaak word in die volledige spesifikasie wat by die Patentkantoor onder bovermelde nommer ingedien is en waarvan 'n

Form P.2; afskrif aangeheg is tesame met die betrokke Vorm P. 2;

NOW THEREFORE these Letters Patent are to grant to the Patentee a patent, the effect of which shall be to grant to the SO IS DIT dat hierdie Patentbrief aan die Patenthouer 'n patent verleen wat die uitwerking het dat, behoudens die

Patentee in the Republic, subject to the provisions of the Act, for the duration of the patent, the right to exclude other persons bepatings van die Wet, aan die Patenthouer vir die duur van die patent in die Republiek die reg verleen word om ander

from making, using, exercising or disposing of the invention, so that he shall have and enjoy the whole profit and advantage persone uit te sluit van die vervaardiging, aanwending, uitoefening of van die handsetting van die uitvinding, sodat hy al die

accruing by reason of the invention. wins en voordeel wat uit die uitvinding voortspruit, verkry en geniet.

IN TESTIMONY WHEREOF the seal of the Patent Office has been affixed at Pretoria with effect from the TER BETUIGING WAARVAN die seël van die Patentkantoor hierop te Pretoria aangebring is met ingang van die

24th day of APRIL nineteen hundred and NINETY SIX

Registrar of Patents • Registrateur van Patente

[See overleaf [Blaai om



## **PATENT**

- 1. The patent remains in force only if the required renewal fees are paid annually after the third year of the date of application. Die patent is geldig slegs indien die vereiste hernuwingsgelde jaarliks, na die derde jaar vanaf die datum van aansoek betaal word.
- 2. This document is not valid unless sealed with the seal of the Patent Office, PRETORIA. Hierdie dokument is nie geldig nie tensy geseël met die seël van die Patentkantoor, PRETORIA.



## John & Kernick

Patent Attorneys and Trade Mark Agents Attorneys, Notaries and Conveyancers

NOTES

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Att: Dr S.A. Allison

Randburg

2125

25th May, 1996

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Our Reference:

12403 ZA /MVS

Your Reference:

17/0/171

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SOUTH AFRICAN PATENT APPLICATION NO. 95/4458 entitled THE RECOVERY OF METAL VALUES FROM SLAGS. (Based on your earlier S.A. Provisional Application No. 94/3729)

Dear Sir / Madam

We have much pleasure in enclosing the Letters Patent Document issued in respect of the above application the particulars of which are as follows :-

PATENTEE

MINTEK

PRIORITY CLAIMED

94/3729

10th June, 1994

FILING DATE

31st May, 1995

TERM OF PATENT

Twenty years from the date of filing

PATENT EXPIRES

31st May, 2015

RENEWAL FEES

Due annually starting three years

after the date of filing

N.B. While we do make a practice of sending reminders in regard to the dates on which renewal fees become due for payment cannot accept any responsibility for failure to do so or for the fact that a reminder may not be received by the due date.

Yours faithfully

KERNICK.

	FRICA	REGISTER OF PATEN	TS	PATENTS ACT, 197	
OFFICIAL APPLICATION	NO.,	LODGING DATE: PROVISIONA	L	ACCEPTANCE DATE	
21 01	15/4458	22		47 13-2-96	
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		, Glen Michael DENTO			
Derek Ala	COUNTRY	, Glen Michael DENTO	DATE	· · · · · · · · · · · · · · · · · · ·	
Derek Ala				10th June, 1994	
PRIORITY CLAIMED  N.B. Use International abbreviation for country	COUNTRY	NUMBER 94/3729	DATE	•	
PRIORITY CLAIMED  N.B. Use International abbreviation for country (See Schedule 4)  TITLE OF INVENTION	COUNTRY  ZA  33  RECOVERY	94/3729 31 OF METAL VALUES FF	DATE	10th June, 1994	
PRIORITY CLAIMED  N.B. Use International abbreviation for country (See Schedule 4)  TITLE OF INVENTION  54  THE  ADDRESS OF APPLICANT	COUNTRY  ZA  33  RECOVERY  (S)/PATENTEE  Strijdom	94/3729 31 OF METAL VALUES FF	DATE 32 ROM SLA	10th June, 1994	
PRIORITY CLAIMED  N.B. Use International abbreviation for country (See Schedule 4)  TITLE OF INVENTION  54  THE  ADDRESS OF APPLICANT	COUNTRY  ZA  33  RECOVERY  (S)/PATENTEE  Strijdom	NUMBER  94/3729  31  OF METAL VALUES FF  (S)  Drive, Randburg, T	DATE 32 ROM SLA	10th June, 1994	
PRIORITY CLAIMED  N.B. Use International abbreviation for country (See Schedule 4)  TITLE OF INVENTION  54 THE  ADDRESS OF APPLICANT  200 Hans S Province,	COUNTRY  ZA  33  RECOVERY  (S)/PATENTEE  Strijdom Republic	NUMBER  94/3729  31  OF METAL VALUES FF  (S)  Drive, Randburg, T	DATE 32 COM SLA	10th June, 1994	
PRIORITY CLAIMED  N.B. Use International abbreviation for country (See Schedule 4)  TITLE OF INVENTION  54 THE  ADDRESS OF APPLICANT  200 Hans Service,  ADDRESS FOR SERVICE  74 JOHN & KERNICK, V.	COUNTRY  ZA  33  RECOVERY  (S)/PATENTEE  Strijdom Republic	94/3729 31 OF METAL VALUES FF  (S) Drive, Randburg, Telephone of South Africa.	DATE 32 COM SLA	10th June, 1994 AGS.	
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## John & Kernick FORM P7

REPUBLIC OF SOUTH AFRICA PATENTS ACT, 1978

## **COMPLETE SPECIFICATION**

(Section 30(1) - Regulation 28)

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21	01 Official application No.	22 Lodging date	J&K Reference
_	954458	31st May, 1995	P 12403 ZA/MVS
51	International classification		
	C22B		
71	Full Name(s) of Applicant(s)		
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72	Full name(s) of Inventor(s)		
	Derek Alan HAYM	IAN, Glen Michael DENTON.	
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	THE RECOV	ERY OF METAL VALUES FROM S	STAGS.
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#### THE RECOVERY OF METAL VALUES FROM SLAGS

#### FIELD OF THE INVENTION

This invention relates to the recovery of metal values from slags, and, more particularly, from slags which are produced in matte production processes, such as those produced in the treatment of a concentrate for the recovery of nickel and copper therefrom. Still more particularly, but not exclusively, the invention is concerned with the recovery of cobalt from slags produced in matte production processes. This includes both the slag product in the production of a green

/matte, and

matte, and the converter slags produced in the subsequent treatment of a green matte to produce a white matte.

## BACKGROUND TO THE INVENTION

Sulphide mattes are produced in many different metal recovery processes and, most particularly, in the production of nickel and copper from flotation or other concentrates containing same.

Generally, a green matte is firstly produced. The slag from the production of such green matte is often discarded. There are thus vast quantities of such slags in dumps at certain plants.

The green matte is then blown in a converter to produce a white matte and another slag, herein termed the converter slag.

Both slags, but in particular the converter slag, often contain a significant quantity of cobalt, in addition to certain amounts of copper, nickel and, usually, significant proportions of iron.

/The converter

The converter slag is usually recycled to the furnace in which the green matte is produced and some of the metal values contained therein are recovered, whilst a substantial proportion is lost. This process also has the disadvantage that the oxides are recycled to the sulphide treatment step thereby re-mixing the sulphide and oxide cycles.

#### OBJECT OF THE INVENTION

It is the object of this invention to provide for the improved recovery of metal values contained in slags emanating from matte production processes and, in particular, but not exclusively, from a converter slag.

#### SUMMARY OF THE INVENTION

In accordance with this invention there is provided a process for the recovery of metal values from a feed slag which contains metal values and which emanates from a matte production process, the process comprising sub-dividing the feed slag and charging it into an open arc d.c. furnace having an elevated electrode and a molten bath acting as the

other electrode, or charging the feed slag in a molten state into the furnace, treating the feed slag in the furnace with carbonaceous reductant to form a metal phase and a slag phase, recovering the metal phase, and treating the metal phase for the recovery of the desired metal values therein.

Further features of the invention provide' for the feed slag to be a converter slag produced in a nickel and copper recovery process during blowing of a green matte to produce a white matte; for the amount of carbonaceous reductant employed to be less than the stoichiometric amount required to reduce all metal oxides in the feed slag to metal and, more particularly, for the amount carbonaceous reductant to be chosen so as to limit the reduction of iron oxides present in the feed slag to thereby enhance the proportion of other metal values, in particular cobalt and, most particularly, for the amount of carbonaceous reductant employed to be from 30% to 60% of said stoichiometric amount; for the reduction to take place at a temperature of at least 1400° C and preferably 1500 - 1600° C; for the carbonaceous reductant and feed slag to be either fed simultaneously to the furnace or, more preferably,

for the feed slag to be pre-melted prior to feeding of the carbonaceous reductant to the furnace; and for the molten bath to be employed as the anode in the d.c. electrical circuit.

The invention still further provides for the metal produced in the reduction process to be leached, conveniently with spent electrolyte from a copper or nickel electrowinning process; and for iron to be precipitated out of the resultant leach solution by decreasing the pH appropriately. The cobalt, copper and nickel can then be separated from each other and recovered using any suitable hydro-metallurgical process.

The invention also provides a matte production process in which a green or a white matte is produced in a smelter and the resultant slag is treated by a process defined above.

In order to test the invention, various experiments were carried out and, in order that the invention may be more fully understood, certain of such experiments will be described below.

# DETAILED DISCUSSION OF THE INVENTION AND EXPERIMENTAL TEST RESULTS

In experimental tests conducted in order to initially demonstrate operation of the invention, a converter slag sample, which had certain inclusions of matte therein, was treated according to the invention. The slag and matte had the following compositions:

#### ANALYSIS OF THE RAW MATERIALS

Analyses (mass %)							,		
	Co	Cu	Ni	Fe#	Si02	MqO	A1203	CaO	S
	0.8-	0.35-	1.0-						
Slag	1,1	0.45	1.2	48.5	30.4	0.5	0.3	< 0.1	-
Matte	1.75	13.2	47.4	8.5	-	-	- "	-	24.4

#Total iron

The carbonaceous reductant was a sub-divided coal which had the following analysis:

#### COAL ANALYSIS

Proximate analysis	
Moisture, %	0.8
Volatiles, %	25.0
Ash, %	11.9
Fixed carbon, % (by diff.)	62.3
Ash analysis	
Total silica, as SiO2 %	42.6
Aluminium, as Al203 %	26.7
Total iron, as Fe203 %	15.3
Calcium, as CaO %	6.27
Magnesium, as MgO %	0.25
Sulphur	2.3
Total	102.3

The tests were conducted in a 200kVA open arc furnace which employed the molten bath as the anode, and a elevated single, electrode with feeding effected from the top of the furnace. In order to commence operation of the furnace an 8 hour warm up period was employed during 10kg of converter matte and 80kg of converter slag were melted down. At the end of the warm up period the heel material was tapped, after which a number of batches of converter slag and reductants were treated with different parameters and the following results were achieved. this purpose as much of the matte as reasonably possible was removed from the slag by hand.

Operating at a total power of 100kW (made up of a heat loss estimate of 55kW and a specific energy requirement of 0.15kWh/kg of feed) and an operating temperature of 1509°C an alloy was produced having an analysis of

4,62% cobalt

6,74% copper

28,55% nickel, and,

44,27% iron.

The slag, on the other hand, contained 0,28% cobalt

0,38% copper, and

0,40% nickel

Feeding of the slag and reductant were carried out simultaneously and the amount of carbonaceous reductant used was 6% by weight of the converter slag, i.e. 34,3% of the stoichiometric amount which would have been required to reduce all the metal oxides present in the slag.

In a second series of tests in which the coal addition was increased to 8% (45,7% of stoichiometric) and the specific energy requirement raised to approximately 0,55kWh/kg of feed, the average tapping temperature was 1489°C.

The alloy produced had a composition of

cobalt 4,23%,

copper 5,74%,

nickel 24,52%, and,

iron 50,86%.

The slag contained

cobalt 0,23%

copper 0,36%, and,

nickel 0,31% respectively.

It is to be noted that the increase in reductant resulted in an increased dilution by the iron.

In a subsequent series of tests 6% coal (34,3% of stoichiometric) was added only after the slag addition had been melted down over a period of a half an hour. The tapping temperature was 1465°C and the alloy produced had a composition of

5,18% cobalt,

7,91% copper,

34,17% nickel, and,

35,61% iron.

The slag contained

0,29% cobalt

0,35% copper, and

0,33% nickel.

A still further series of tests employed 8% by weight of the coal and, in this case, the tapping temperature was even lower at 1412°C. The alloy produced contained

5,52% cobalt,

8,34% copper,

34,9% nickel, and,

36,01% iron

/The slag

The slag contained

cobalt 0,25%

copper 0,30%, and,

nickel 0,17%.

Larger scale tests were then carried out in a 500kW open arc DC furnace. In all cases these runs were carried by melting the slag first and adding the carbonaceous reductant to the molten slag subsequently. The reason for this is that it is envisaged that in practice, molten slag from a matte production process will immediately be treated according to the invention while still in its molten, or at least hot, state.

In each case, accordingly, the sub-divided solid slag was firstly fed to a molten bath in the preheated furnace at a rate automatically controlled to ensure that the feed and energy inputs were balanced and to provide a constant temperature of operation. After the batch of slag had been added to the furnace the sub-divided coal was then added over a period of time under similar conditions.

/ In this

In this case the slag had an average composition of 0,93 mass percent Co; 0,49 mass percent Cu; 1,47 mass percent Ni; 50,8 mass percent Fe; 29,2 mass percent SiO<sub>2</sub>; 0,18 mass percent CaO; 0,22 mass percent MgO; 0,4 mass percent Al<sub>2</sub>O<sub>3</sub>; and 1,07 mass percent S.

The coal analysis, on average, had a moisture content of 0,2 mass percent; 26,2 mass percent volatiles; 11,35 mass percent ash; and 62,45 mass percent fixed carbon.

Runs were carried out under five different conditions and the results, on the basis of cobalt recovery, are set out below in Tables 1(a) and 1(b).

### MAIN FACTORS AFFECTING COBALT RECOVERY - TABLE 1(a)

CONDITION	BATCH MASS (kg)	MASS COAL BY WEIGHT	% of STOICHIOMETRIC	COBALT RECOVERY
. 1	500	8	43,5	75,0
2	500	9	49,0	71,3
3	500	11	59,9	69,5
4	1000	11	59,9	81,8
5	1000	9	49,0	86,1

/ MAIN FACTORS

#### MAIN FACTORS AFFECTING COBALT RECOVERY - TABLE 1(b)

CONDITION	MELT DURATION (min)	REDUCTION DURATION (min)	TOTAL TIME (min)	MELT TEMP °C	TAP TEMP °C
1	124,0	91,1	215,1	1520	1495
2	65,7	86,2	151,9	1528	1514
3	60,6	85,2	145,8	1498	1454
4	141,0	144,0	298,3	1543	1314
5	115,5	150,5	266,0	1407	1375

It will be noted that coal additions of at least 9% (49,0% of stoichiometric) gave cobalt recoveries of greater than 80% under suitable conditions.

It will be understood that numerous different parameters and operating conditions may be used without departing from the scope of the invention. Also, the invention is not limited to the recovery of metals identified above. Also, the slag may be a slag produced in the green matte production furnace.

- A process for the recovery of metal values l. from a feed slag which contains metal values and which emanates from a matte production process, the process comprising sub-dividing the feed slag and charging it into an open arc d.c. furnace having an elevated electrode and a molten bath acting as the other electrode, or charging the feed slag in a molten state into the furnace, treating feed slag in the furnace carbonaceous reductant to form a metal phase and a slag phase, recovering the metal phase, and treating the metal phase for the recovery of the desired metal values therein.
- 2. A process as claimed in claim 1 in which the feed slag is a converter slag produced in a nickel and copper recovery process during blowing of a green matte to produce a white matte.

- 3. A process as claimed in either of claims 1 or 2 in which the amount of carbonaceous reductant employed is less than the stoichiometric amount required to reduce all metal oxides in the feed slag to metal.
- 4. A process as claimed in claim 3 in which the amount of carbonaceous reductant is from 30% to 60% of said stoichiometric amount.
- 5. A process as claimed in any one of the preceding claims in which the reduction takes place at a temperature of at least 1400° C.
- 6. A process as claimed in claim 5 in which the said temperature is from 1500°C to 1600°C.
- 7. A process as claimed in any one of the preceding claims in which the carbonaceous reductant and feed a slag are fed simultaneously to the furnace.
- 8. A process as claimed in any one of claims 1 to 6 in which the carbonaceous reductant is fed to the furnace subsequent to the feed slag being premelted therein.

- 9. A process as claimed in any one of the preceding claims in which the molten bath is employed as the anode in the d.c. electrical circuit.
- 10. A process as claimed in any one of the preceding claims in which the metal produced in the reduction process is leached followed by decreasing of the pH to precipitate iron.
- 11. A process as claimed in claim 10 in which leaching is effected using spent electrolyte from a copper or metal electrowinning process.
- 12. A composite process comprising a matte production process in which either white or green matte is produced in a smelter and the resultant slag is treated as the feed slag by a process as claimed in any one of claims 1 to 11.
- 13. A process for the recovery of metal values from a feed slag substantially as herein described and exemplified.

DATED on this the 31ST DAY OF MAY 1995.

JOHN & KERNICK for the Applicant AMENDMENTS DATED THIS 29TH DAY OF AUGUST 1996.

> JOHN & KERNICK FOR THE APPLICANT