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(54) Title: ONE STEP PROCESS FOR PRODUCTION OF NI AND FE-NI USING MICROWAVE ASSISTED NON THERMAL HYDROGEN PLASMA

(57) Abstract: The present invention relates to a one step environment friendly process for the preparation of Ni and Fe-Ni has been established, wherein the said process involves highly reactive low temperature non thermal hydrogen plasma for the production of nickel and ferronickel. Reduction reactions in plasma state are spontaneous due to low gibbs free energy and higher reducing potential of the active species in plasma. Atoms and ions of hydrogen are 3 to 15 times respectively more reactive than gaseous hydrogen molecules. The present invention gives the possibility of utilisation of plasma route to produce nickel and ferronickel. This process is eco-friendly since it is free from any carbonaceous reducing source. The by-product of the process is water, which is benign.

ONE STEP PROCESS FOR PRODUCTION OF Ni and Fe-Ni USING MICROWAVE ASSISTED NON THERMAL HYDROGEN PLASMA

FIELD OF INVENTION:

The present invention relates to a process of reduction of NiO to pure metallic Ni and production of Fe-Ni in one step by solid state reduction of a mixture of iron oxide and nickel oxide in the presence of non thermal hydrogen plasma.

BACKGROUND OF THE INVENTION:

Two major sources for extraction of Nickel are lateritic and sulphide deposits. Nickel is used as one of the major constituents of the nickel steel, non ferrous alloys and super alloys. Similarly, ferro-alloys also find application for production of stainless steel, therefore, indicating a strong relationship between ferro-alloy and steel making sector. Pyrometallurgical and hydrometallurgical routes are used for extraction of Ni from lateritic ores and pyrometallurgical route is used for extracting Ni from sulphide ore. Conventional ferronickel production process is based on Rotary Kiln – Electric Furnace Technology (RKEF). It can be in shielded arc as well as submerged arc mode. Electric arc furnaces are employed for nickel above 8%. Reference may be made to Rodeney T Jones, "Smelting applications of DC arc furnaces", Mintek, 200 Malibongwe drive, Private Bag X3015, Randburg, 2125, South Africa, wherein technology for producing unrefined FeNi from the lateritic ore deposits using DC arc furnace has been developed. In this process, Ni containing lateritic material along with the carbonaceous reducing agent is fed into the molten bath. The feed material is dried and calcined before putting in the furnace. The process is applicable to a wide composition range of the feed to effectively produce different grades of ferronickel simultaneously achieving excellent extraction of Ni from slag (the residual Ni is less than 0.2%) [Reference may be made to I. J. Kotze, "Pilot plant production of ferronickel from Ni oxide ores and dusts in DC arc furnace", Minerals Engineering,15, 2002, pp. 1017–1022]. It has the advantage that the dusty material generated during the pre treatment process which cannot be charged in the conventional arc furnace without agglomeration can be processed directly into the DC arc furnace, thereby improving overall recovery.

Use of hydrogen plasma has not been explored or studied for production of nickel/ferronickel. The process involves highly reactive low temperature non thermal hydrogen plasma for the production of nickel and ferronickel. Reduction reactions in plasma state are spontaneous due to low gibbs free energy and higher reducing potential of the active species in plasma. Atoms and ions of hydrogen are 3 and 15 times respectively more reactive than gaseous hydrogen molecules. The present invention gives the possibility of utilisation of plasma route to produce nickel and ferronickel. This process is eco-friendly since it is free from any carbonaceous reducing source. The by-product of the process is water, which is benign.

The current invention opens up several possibilities as listed below:

1. Unlike conventional process of nickel and ferronickel production, the non-thermal plasma process involves only single step.

2. The size of the reactor is small.

3. Since there is no involvement of carbon/coke, the process is eco friendly.

4. The water generated as a by-product can be recycled to produce hydrogen, which is another advantage.

5. The atomic, ionic and excited species in plasma state drives the reduction reaction very fast, making the process less time taking.

6. Zero carbon dioxide/monoxide emission will attract carbon credit.

Nickel oxide reduction with hydrogen gas is already studied by few researchers. The production of nickel powder from leach solution by hydrometallurgical route under pressure using hydrogen and influence of all the parameters is reviewed by Agrawal et. al. [A. Agrawal, V. Kumar, B. D. Pandey, K. K. Sahu, " A comprehensive review on the hydrometallurgical processes for the production of nickel and copper powders by hydrogen reduction", Material Research Bullitin, 41, 2006, pp. 879 – 892] and effect of the operating parameters like pH, concentration of metals, particle size of additives and operating

conditions of the autoclave on reduction and the powder produced are well explained. Reference may be made to J. T. Richarson, R. Scates, M. V. Twigg, " X-ray diffraction study of nickel oxide reduction by hydrogen", Applied Catalysis A: General 246, 2003, pp. 137 – 150, wherein Richardson has studied NiO reduction with the help of a hot stage XRD within a temperature range of 175 - 300°C. Another attempt was made by Utigard et. al. [T. A. Utigard, M. Wu, G. Plascencia, T. Marin, " Reduction kinetics of goro nickel oxide using hydrogen", Chemical Engineering Science, 60, 2005, pp. 2061 – 2068] to study the kinetics of Goro Nickel using thermogravimetery.

Reference may be made to US 4,398,945, wherein iron oxides are first reduced and carburised to iron carbides with simultaneous disruption of ferrite structure of nickel ferrites. This carburised product can directly be fed into electric arc furnace to produce ferronickel product. The processes avoid addition of ferrosilicon. The remaining iron carbide and nickel is also recovered due to the magnetic properties of ferronickel.

Reference may be made to US 8,728,195 B2 and P. Rajput, B. Bhoi, S. Sahoo, R. K. Paramguru, B. K. Mishra, "Preliminary Investigation into Direct Reduction of Iron in a Low Temperature Hydrogen Plasma, 40, 2013 , pp. 61-68, wherein a process of producing direct reduced iron using hydrogen plasma. It was shown that the partial pressure of the reductant is much lesser when it is atomic, ionic and excited species compared to the molecular gas. Also, it was demonstrated that iron can be produced at a lower temperature in presence of plasma. It is by virtue of the highly reactive species that the activation energy in plasma state reduces, and the process can be achieved at a lower temperature and partial pressure conditions.

OBJECTIVE OF INVENTION:

The main objective of the present invention is to develop a process to produce Ni metal and ferro nickel alloy in one step from NiO and synthetic mixture of Ni and Fe oxide using non thermal microwave assisted hydrogen plasma.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1: XRD pattern of the raw material (a) Nickel oxide (b) Ferronickel

Figure 2: Schematic of reactor chamber. (1) hydrogen gas inlet, (2 and 3) water inlet and outlet respectively, (4) hydrogen plasma, (5) sample holder, (6) Quartz ring, (7) microwave power supply (6kW), (8) vacuum, (9) copper plate, (10) sample pellet, (11) pyrometer.

Figure 3: XRD pattern of the reduced sample; example 1

Figure 4: SEM image of the representative reduced sample along with the Ni dispersion

Figure 5: XRD pattern of the reduced sample; example 5

Figure 6: XRD pattern of the reduced sample; example 3

Figure 7: XRD pattern of the reduced sample; example 4

Figure 8: XRD pattern of the reduced sample; example 5

BRIEF SUMMARY OF THE INVENTION:

Accordingly the present invention provides a process for the production of Ni and Ferronickel in single step which comprises:

(a) Preparation of NiO and (25% NiO + Fe_2O_3) pellets as raw material.

(b) Loading of raw material into the reaction chamber.

(c) Evacuation of chamber and processing of the raw pellet at the hydrogen flow rate of 70 – 250 sccm, chamber pressure between 5 – 50 Torr, time of reduction 5 – 20 min at constant power supply for NiO and hydrogen flow rate of 70 – 250 sccm, pressure ranging between 5 – 30 Torr, time of reduction was varied from 5 - 180 min at constant power supply for a mixture of Fe_2O_3-NiO, cooling of the sample to obtain desired Ni and Ferronickel.

In an embodiment of the present invention, the process described here is environmental friendly since no carbonaceous source is used.

In another embodiment of the present invention, Ni and Ferronickel produced is devoid of Carbon, Sulphur, and Phosphorus.

In another embodiment of the present invention, the reaction in single step process is being carried out in highly reactive species of Hydrogen plasma.

DETAILED DESCRIPTION OF THE INVENTION:

Currently pyrometallurgical and hydrometallurgical routes are used for extraction of Ni from lateritic ores and pyrometallurgical route is used for extracting Ni from sulphide ore. The Ni is then used for production of Ferronickel in a DC arc furnace or a rotary kiln. The present invention utilises low temperature non thermal hydrogen plasma as a reductant as well as the heating source. Hydrogen plasma consist of highly active species like hydrogen ions, hydrogen atoms and excited hydrogen molecules, which drive the reaction very fast thereby decreasing the residence time for reduction reaction to take place. After the oxide gets reduced to pure metallic form, alloying takes place in the plasma atmosphere leading to the formation of Ferronickel. The present invention provides a one step process for the production of Ni and Ferro Nickel from its Oxide and a synthetic mixture of Ni and Fe oxide respectively employing a low temperature non thermal hydrogen plasma as given above. This process utilises hydrogen in plasma state as a reductant. The by-product of the process is water which is benign. The process comprises of following steps.

- Raw material in the form of pellets is prepared from oxides.
- Pellets are loaded and the chamber is evacuated to the pressure 10^{-3}mbar.
- Plasma was generated by dissociating the hydrogen gas molecules using standard microwave frequency waves of 2.45GHz.
- For Nickel Oxide, the reduction was carried out at the hydrogen flow rate ranging from 70 – 250 sccm, chamber pressure ranging between 5 -50 Torrat constant power input, the thickness of the sample was varied from 3mm to 9mm, the temperature was within the range of 500 - 1000°C, reduction time was adjusted between 5 – 20 minutes .

- For Fe-Ni sample, the reduction and alloying is carried out at the flow rate ranging from 70 – 250 sccm, chamber pressure ranging between 5 – 30 Torr, at constant power input, thickness of the sample was varied from 3mm to 9mm, the temperature was within the range 500 - 800°C and reduction time was adjusted between 5 – 180 minutes.

- After reduction sample was allowed to cool on its own.

- The reduced sample is then characterised.

The reaction in plasma can be described as:

$H_2 = H + H$

$2H = 2H^+ + 2e^-$

Reduction of NiO in hydrogen plasma:

$NiO + \text{Hydrogen plasma } (2H/2H^+/2H^{2+}/2H^{3+}/H_2^+) = Ni + H_2O(g)$

Reduction of Fe_2O_3 in hydrogen plasma:

Reaction of Fe_2O_3 with atomic hydrogen

$Fe_2O_3 + 6H = 2Fe + 3H_2O\ (g)$

Reaction of Fe_2O_3 with ionic hydrogen:

$Fe_2O_3 + 6H^+ + 6e^- = 2Fe + 3H_2O\ (g)$

The raw materials used in the current study are Fe_2O_3 obtained from Himedia with the minimum assay of 99%, Hydrogen gas which is 99.999% pure.

Laboratory grade NiO oxide is compacted in the form of pellets and is directly used as a raw material; for ferronickel, Fe_2O_3 and 25% NiO powder by weight is mixed using a

mortar pestel and pellets were prepared from it. The compacted Iron and Nickel oxide pellets were used as raw material for producing ferronickel. The XRD pattern of the NiO and the raw material for ferronickel is presented in figure 1(a) and 1(b) respectively. Total Ni in NiO is estimated 64.44% by wet chemical analysis. Chemical analysis details of the raw material for ferronickel production are given in Table 2. (Total iron: Fe(T), Metallic iron: Fe(M))

Table 2. Chemical analysis of raw material for ferronickel

Phase/%	**Fe(T)**	**Fe(M)**	**FeO**	**Ni**
	51.10	0.56	0.74	16.73

Brief description of the drawings:

Figure 2 gives the schematic of the reactor chamber. (1) hydrogen gas inlet, (2 and 3) water inlet and outlet respectively, (4) hydrogen plasma, (5) sample holder, (6) Quartz ring, (7) microwave power supply (6kW), (8) vacuum, (9) copper plate, (10) sample pellet, (11) pyrometer.

The examples to illustrate the invention are presented below, example 1-3 describe the production of Ni metal and example 4 and 5 describe the production of ferronickel using low temperature non-thermal hydrogen plasma.

Example 1

A microwave plasma reactor of 6kW capacity is used to carry out the experiments. The experimental procedure involves the loading of the sample in the reactor chamber. The sample is kept at the centre of the copper plate such that it must completely be inside the plasma during the experiment. To ensure the purity of plasma the chamber is first evacuated and the vacuum level of 10^{-3} mbar is maintained. The power was supplied and the process parameters were maintained throughout the duration of the process are mentioned below:

Hydrogen flow rate: 250 sccm

Chamber pressure: 35 Torr

Time of reduction: 5 min

Temperature: 800°C

Sample weight before reduction: 2.4025 g

Sample weight after reduction: 1.7311 g

Thickness of sample: 3 mm

The sample was cooled to room temperature and then taken out of the chamber. The reduced sample was then ground and characterised. The XRD pattern of the reduced sample is presented in figure 3. The XRD pattern shows only Ni peaks indicating the formation of pure metallic phase. Also, the weight loss data goes in good agreement with XRD indicating complete reduction of the oxide sample.

The FE-SEM was also done in order to see the microstructure. The SEM image and area mapping through EDAX shown in figure 4 clearly indicated the formation of pure metallic phase and its dispersion. The XRD pattern of the reduced sample is presented in figure 3.

Example 2

NiO sample in the form of pellet is reduced in a microwave assisted hydrogen plasma reactor. The sample is charged into the reactor chamber and plasma was switched ON. The process parameters are listed below:

Hydrogen flow rate: 150 sccm

Chamber pressure: 35 Torr

Time of reduction: 5 min

Temperature: 1000°C

Sample weight before reduction: 7.0520 g

Sample weight after reduction: 5.6456 g

Thickness of sample: 9 mm

The reduced sample is cooled to room temperature and is taken out of the chamber. The sample is then ground and characterised. The XRD pattern of the reduced sample is shown in figure 5. The chemical analysis indicated the presence of 85% metallic Ni in the reduced sample. Percentage weight loss was recorded to be 19.94%.

Example 3

NiO sample in the form of pellet is reduced in a microwave assisted hydrogen plasma reactor. The sample is charged into the reactor chamber and plasma was switched ON. The process parameters are listed below:

Hydrogen flow rate: 400 sccm

Chamber pressure: 52 Torr

Time of reduction: 10 min

Temperature: 1100˚C

Sample weight before reduction: 7.4467 g

Sample weight after reduction: 5.1684 g

Thickness of sample: 9mm

After the experiment, the sample was allowed to cool. The reduced sample is then ground into fine powder and is characterised. XRD is used for phase analysis and the quantitative estimation is made following the wet chemical procedure. Although the % weight loss recorded gives 100% reduction of the sample, the volumetric titration of the sample indicated around 92% reduction (91.76% Ni metal). This variation could be due to the loss of sample when plasma struck.

Example 4

The pellets prepared by mixing the oxides of iron and nickel(25% Ni) are used as a raw material for producing ferronickel. The experimental setup used is a microwave assisted plasma reactor. Hydrogen is used as a plasmagen gas. The sample was charged and then the reactor chamber was evacuated. Plasma was started and sample was reduced as per the process parameters listed below.

Hydrogen flow rate: 70 sccm

Chamber pressure: 14 Torr

Time of reduction: 180 min

Temperature: 800˚C

Sample weight before reduction: 2.5124 g

Sample weight after reduction: 1.9250 g

Thickness of sample: 3 mm

The sample was allowed to cool till room temperature and is then taken out from the reaction chamber. The percentage reduction in weight after processing was found to be 23.33%. It is ground and characterised using XRD and wet chemical analysis procedures. The XRD pattern of the reduced sample is shown in figure 7. It shows the presence of metallic Fe and Ni along with the alloyed phase of iron and nickel. The chemical analysis result is presented in Table 3.

Table 3. Chemical analysis of the reduced sample

Phase/%	**Fe(T)**	**Fe(M)**	**FeO**	**Ni as NiO**
	60.41	22.58	28.91	26.28

Example 5

The pellets prepared by mixing the oxides of iron and nickel(25% Ni) are used as a raw material for producing ferronickel. The experimental setup used is a microwave assisted plasma reactor. Hydrogen is used as a plasmagen gas. The sample was charged and then the

reactor chamber was evacuated. Plasma was started and sample was reduced as per the process parameters listed below.

Hydrogen flow rate: 70 sccm

Chamber pressure: 14 Torr

Time of reduction: 30 min

Temperature: 800°C

Sample weight before reduction: 2.5596 g

Sample weight after reduction: 1.1907 g

Thickness of sample: 3 mm

The sample was allowed to cool till room temperature and is then taken out from the reaction chamber. The percentage reduction in weight after reduction was found to be 14.41%. It is ground and characterised using XRD and wet chemical analysis procedures. The XRD pattern of the reduced sample is shown in figure 8. It shows the presence of metallic Fe and Ni along with the alloyed phase of iron and nickel. The chemical analysis result is presented in Table 4.

Table 4. Chemical analysis of the reduced sample

Phase/%	**Fe(T)**	**Fe(M)**	**FeO**	**Ni as NiO**
	56.16	2.20	39.39	24.55

ADVANTAGES OF THE PRESENT INVENTION:

1. The process described here to produce nickel and ferronickel using low temperature non-thermal hydrogen plasma is eco-friendly.

2. The process eliminated many steps involved in pyrometallurgical and hydrometallurgical route, it is a single step process.

3. The water produced as a by-product can be recycled to generate hydrogen.

4. Since highly reactive species in plasma state take place in the reaction, the reduction reaction is very fast.

5. The reductant used in this process is from a renewable resource.

6. The product is of better quality and purity since there is no source of sulphur, phosphorous etc.

7. The process does not involve any carbon source, hence no carbon dioxide emission.

8. The process can assist in gaining carbon credit.

CLAIMS

1. A process for the production of Ni and Ferronickel in single step which comprises:

   (a) Preparation of NiO and (25% NiO + Fe_2O_3) pellets as raw material.

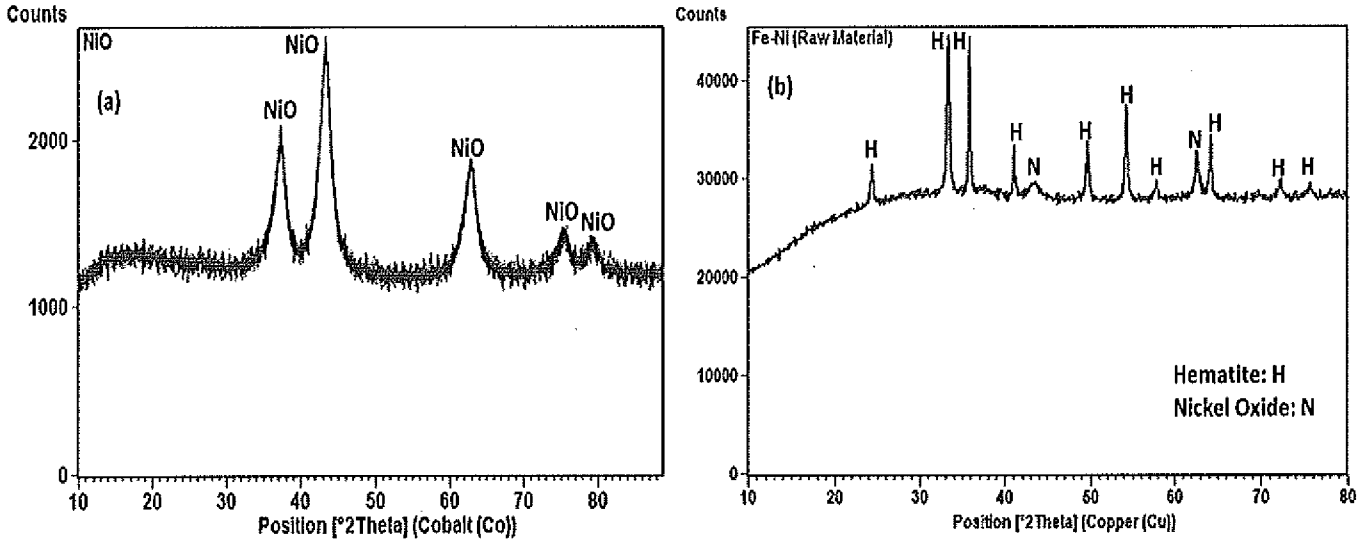
   (b) Loading of raw material into the reaction chamber.

   (c) Evacuation of chamber and processing of the raw pellet at the hydrogen flow rate of 70 – 250 sccm, chamber pressure between 5 – 50 Torr, time of reduction 5 – 20 min at constant power supply for NiO and hydrogen flow rate of 70 – 250 sccm, pressure ranging between 5 – 30 Torr, time of reduction was varied from 5 - 180 min at constant power supply for a mixture of Fe2O3-NiO, cooling of the sample to obtain desired Ni and Ferronickel.

2. The process as claimed in claim 1, is environment friendly since no carbonaceous source is involved.

3. The process as claimed in claim 1, wherein Ni and Ferronickel produced is being devoid of Carbon, Sulphur and phosphorous.

4. The process as claimed in claim 1, wherein the reaction in single step process is being carried out in highly reactive species of Hydrogen plasma.


Counts
NiO
(a)
NiO
NiO
NiO
NiO
NiO
2000
1000
0
10
20
30
40
50
60
70
80
Position [°2Theta] (Cobalt (Co))
Counts
Fe-Ni (Raw Material)
(b)
H
H
H
H
H
H
H
N
N
H
H
H
40000
30000
20000
10000
0
10
20
30
40
50
60
70
80
Position [°2Theta] (Copper (Cu))
Hematite: H
Nickel Oxide: N

(a) (b)

Fig. 1

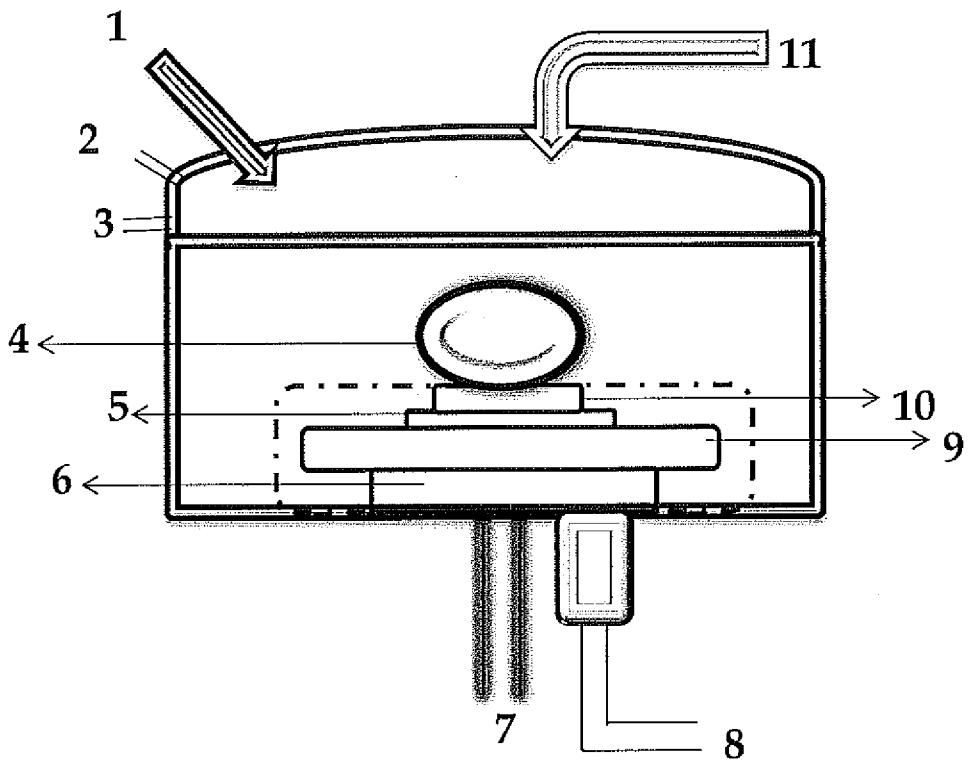


Fig. 2

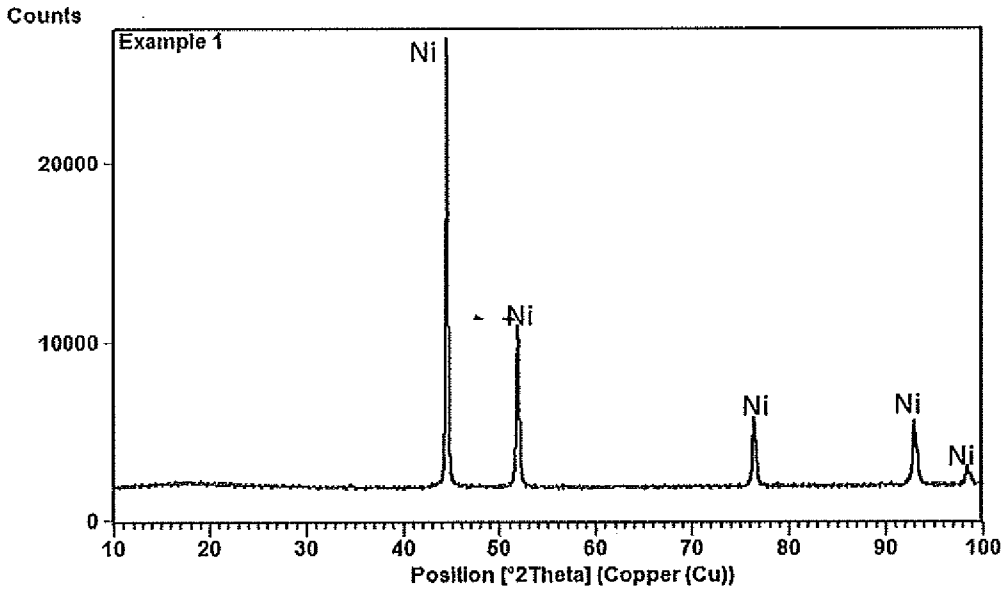

Counts
Example 1
Ni
Ni
Ni
Ni
Ni
20000
10000
0
10
20
30
40
50
60
70
80
90
100
Position [°2Theta] (Copper (Cu))

Fig. 3

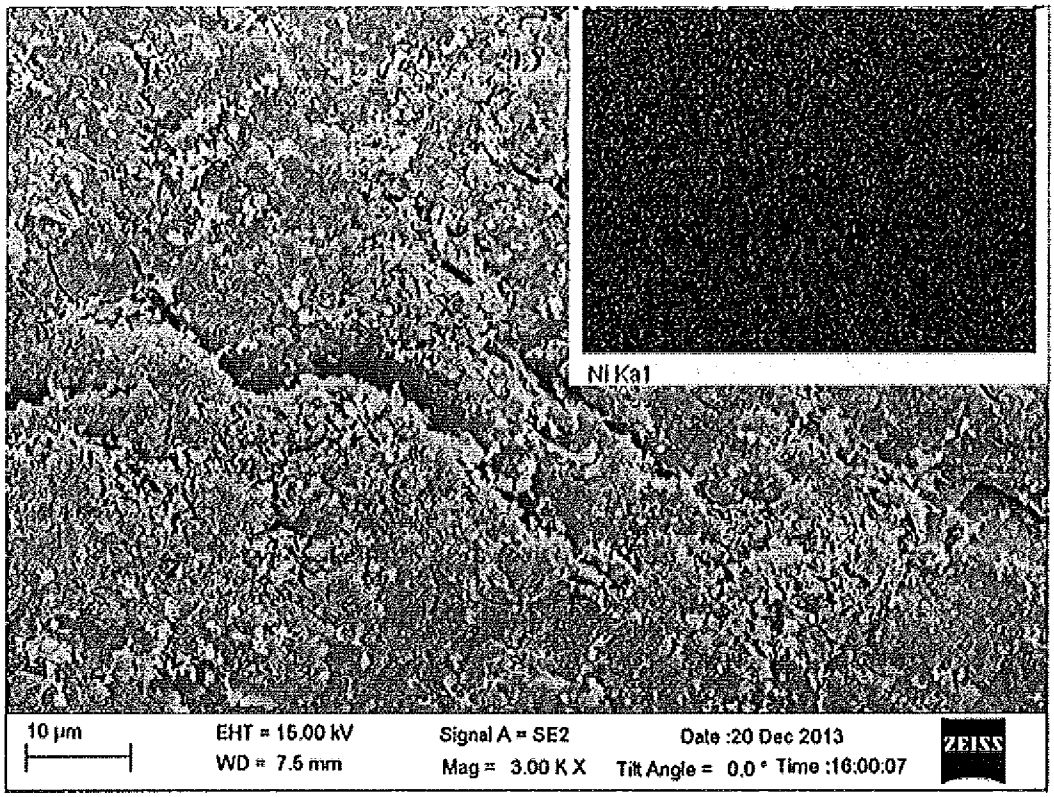

Ni Ka1
10 µm
EHT = 16.00 kV
WD = 7.5 mm
Signal A = SE2
Mag = 3.00 K X
Date :20 Dec 2013
Tilt Angle = 0.0 ° Time :16:00:07
ZEISS

Fig. 4

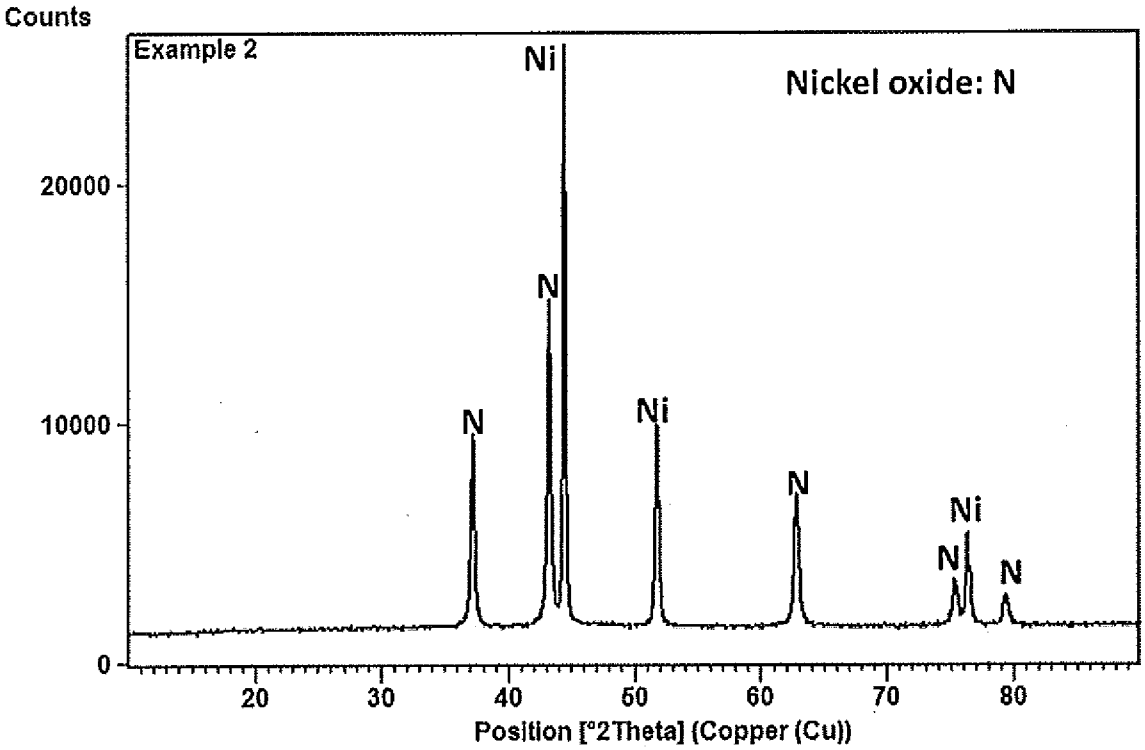

Counts
Example 2
Nickel oxide: N
Ni
N
Ni
N
N
Ni
N
N
20000
10000
0
20
30
40
50
60
70
80
Position [°2Theta] (Copper (Cu))

Fig. 5

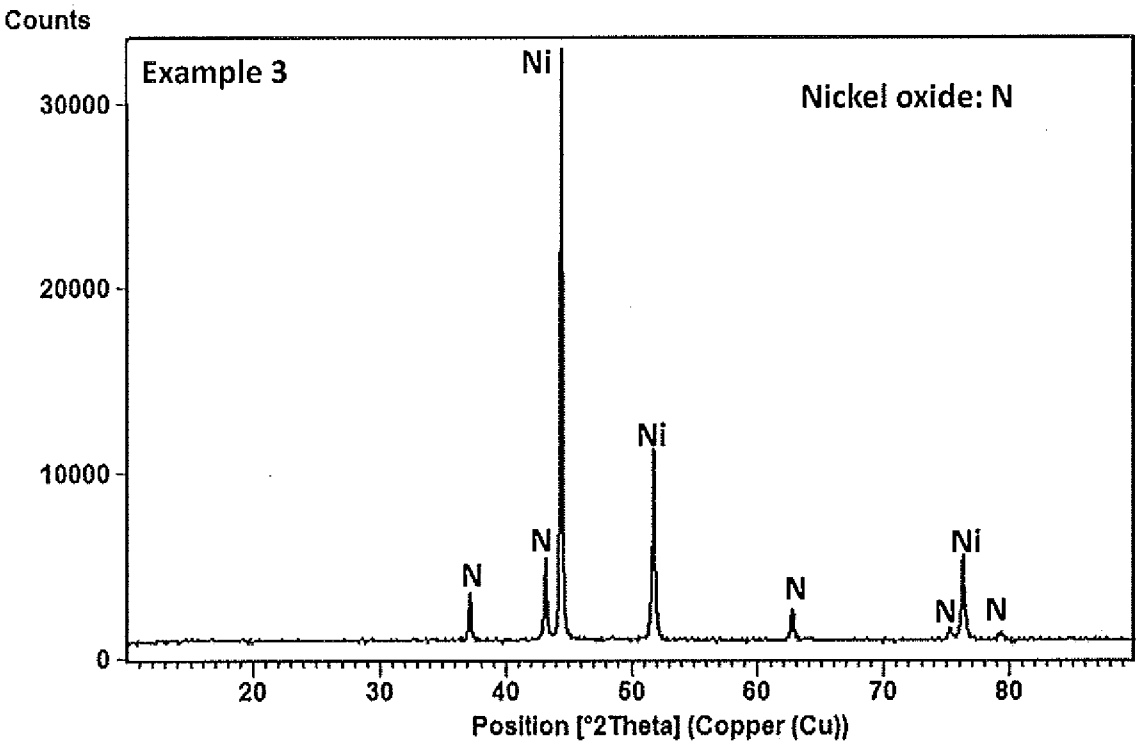

Counts
Example 3
Nickel oxide: N
Ni
Ni
Ni
N
N
N
N
N
30000
20000
10000
0
20
30
40
50
60
70
80
Position [°2Theta] (Copper (Cu))

Fig. 6

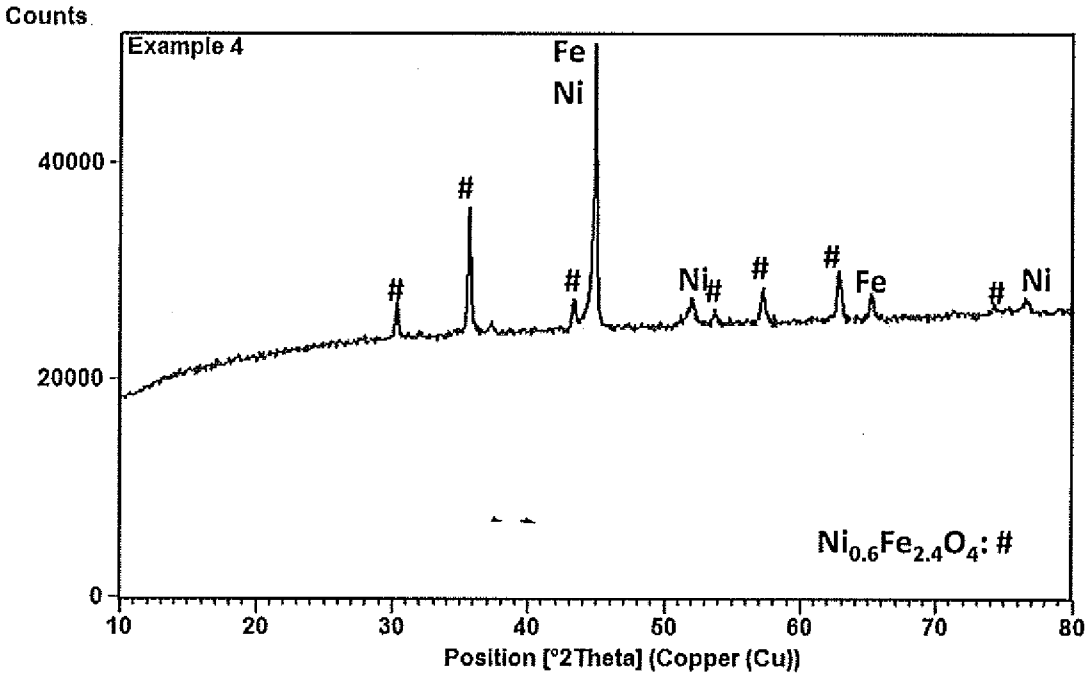

Counts
Example 4
Fe
Ni
40000
20000
0
#
Ni
Fe
10
20
30
40
50
60
70
80
Position [°2Theta] (Copper (Cu))
$Ni_{0.6}Fe_{2.4}O_4$: #

Fig. 7

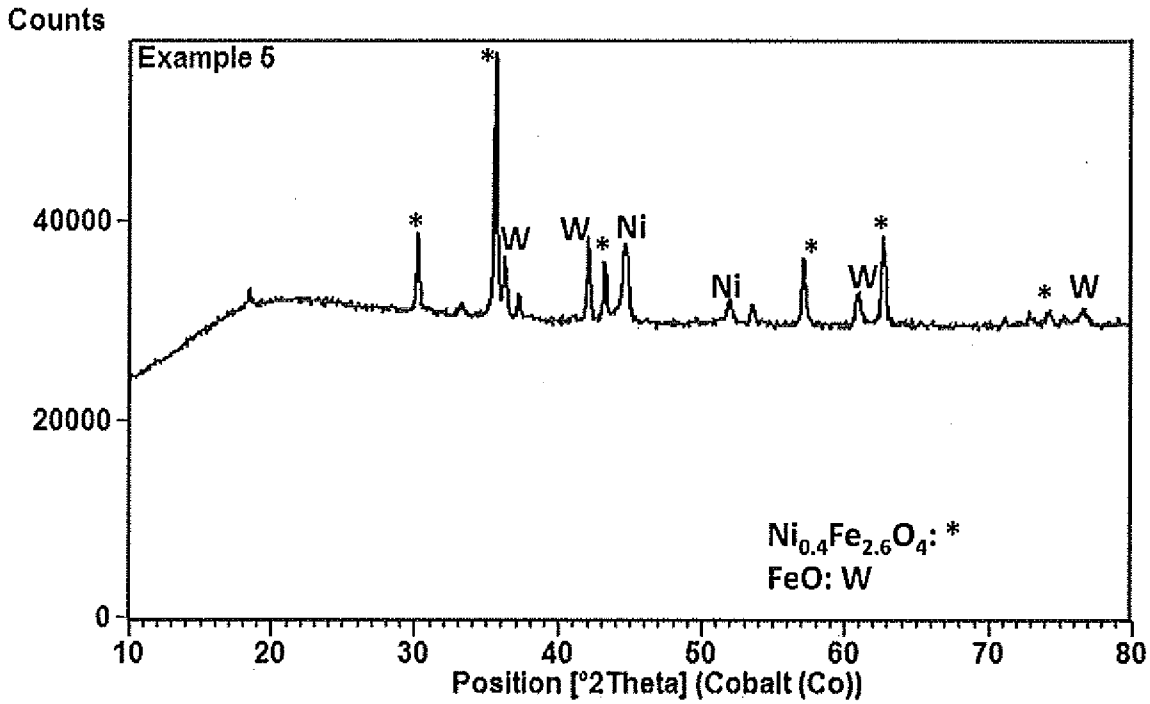

Counts
Example 5
40000
20000
0
10
20
30
40
50
60
70
80
Position [°2Theta] (Cobalt (Co))
Ni0.4Fe2.6O4: *
FeO: W
*
W
Ni

Fig. 8

INTERNATIONAL SEARCH REPORT

International application No
PCT/IN2016/050008

A. CLASSIFICATION OF SUBJECT MATTER
INV. C22B5/12 C22B23/02
ADD.

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C22B

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

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 8 728 195 B2 (BHOI BHAGYADHAR [IN] ET AL) 20 May 2014 (2014-05-20) cited in the application the whole document	1-4
A	HIDAYAT T ET AL: "On the Relationships between the Kinetics and Mechanisms of Gaseous Hydrogen Reduction of Solid Nickel Oxide", METALLURGICAL AND MATERIALS TRANSACTIONS B, SPRINGER-VERLAG, NEW YORK, vol. 40, no. 4, 11 June 2009 (2009-06-11), pages 474-489, XP019735124, ISSN: 1543-1916, DOI: 10.1007/S11663-009-9239-X the whole document	1-4

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[X] Further documents are listed in the continuation of Box C.

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Date of the actual completion of the international search	Date of mailing of the international search report
13 April 2016	21/04/2016

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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	I.J KOTZÉ: "Pilot plant production of ferronickel from nickel oxide ores and dusts in a DC arc furnace", MINERALS ENGINEERING, vol. 15, no. 11, 1 November 2002 (2002-11-01), pages 1017-1022, XP055105152, ISSN: 0892-6875, DOI: 10.1016/S0892-6875(02)00127-9 cited in the application the whole document	1-4
A	US 4 398 945 A (STEPHENS JR FRANK M [US]) 16 August 1983 (1983-08-16) cited in the application the whole document	1-4

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INTERNATIONAL SEARCH REPORT

Information on patent family members

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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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US 4398945 A	16-08-1983	NONE	