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(54) Title: APPARATUS AND METHOD FOR AUTOMATIC CONTROLLING DIRECT REDUCTION PROCESS OF IRON
OXIDE CONTAINING MATERIAL

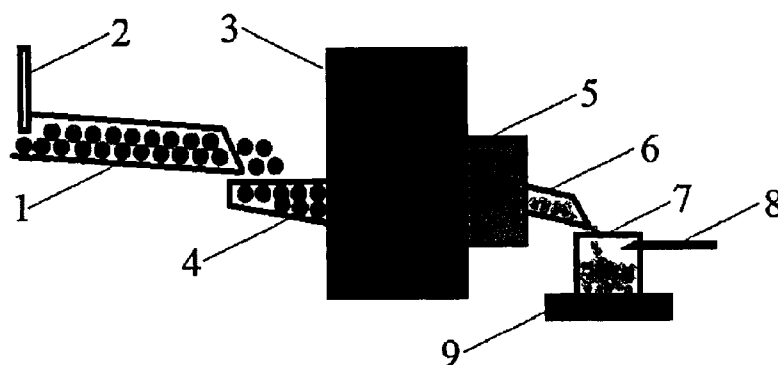


Figure 1

(57) Abstract: The present invention relates to an apparatus and a method for automatic controlling a direct reduction process of an iron oxide containing material. The apparatus comprises sampling means adapted to periodically take samples of metallized pellets during the reduction process of the iron oxide containing material, a mill device for milling the samples of the metallized pellets, density determining means for determining a density of the metallized pellet material in the milled sample and calculation means adapted to estimate the quality of the reduced metallized pellets based on the density determined by the density determining means. The method according to the invention comprises periodically taking samples of metallized pellets during the reduction process of the iron oxide containing material, milling the samples of the metallized pellets to predetermined particle sizes, determining a density of the metallized pellet material in the milled sample and estimating the reduced metallized pellet quality based on the determined density. The use of the apparatus and the method ensures stability of the direct iron reduction process, high level of iron output and product quality.



APPARATUS AND METHOD FOR AUTOMATIC CONTROLLING DIRECT REDUCTION PROCESS OF IRON OXIDE CONTAINING MATERIAL

FIELD OF THE INVENTION

5 The present invention relates to an apparatus and a method for automatic
controlling a direct reduction process of an iron oxide containing material, in particular,
the apparatus and the method according to the present invention can be used for a
precise control the reduction of the pelletized iron oxide containing material in a method
for producing direct reduced iron to ensure the process stability and highest level of iron
10 output.

BACKGROUND OF THE INVENTION

 The Direct Reduction Process for producing metal iron by direct reducing iron
oxide containing materials, known as Midrex process, has become the world's most
successful direct reduction technology. In the direct iron reduction process, metal iron is
15 produced by substantially complete reducing iron oxide containing materials, for
example iron ore in solid phase using a reducing gas typically containing hydrogen and
carbon monoxide which is obtained by reforming a natural gas
(<http://www.midrex.com/uploads/documents/DFM1Q041.pdf>).

 The basic Midrex direct reduction process is described in Begg's patent US
20 3,748,120 for "Method of and apparatus for reducing iron ore oxide to metallic iron"
and US 3,749,386 for "Method and means for reducing iron ore oxides in a gaseous
reduction process". The direct iron reduction process is further developed as disclosed
in many patent documents, for example, in US 6506230 B2 (MIDREX
TECHNOLOGIES INC) for Method for increasing productivity of direct reduction
25 process or in WO 2012/158221 A1 (MIDREX TECHNOLOGIES INC) for System and
method for reducing iron oxide to metallic iron using coke oven gas and oxygen
steelmaking furnace gas.

 The known methods ensures quite high iron content in the reduced iron ore
pellets at the facility output and at present days output process parameters are usually
30 tested with periodic about once per 4 hours by collecting output pelletized ore samples
and laboratory investigation of the output material chemical composition in the samples.
This procedure has been established a lot-of time ago and consists in several

complicated steps involving spectroscopic methods, mass balance calculations etc. These laboratory tests show the quality of the chemical reaction in the ore and after the test the process parameters (like natural gas temperature, natural gas concentration, amount of natural gas, oxygen temperature and etc) can be tuned depending on the output material quality to have the output product of better quality.

The laboratory investigations are precise enough but time consuming and expensive. Besides, there is no information between every two tests. Other weak sides of the known way to check the output material are:

- the way is quite long and poor-automated;
- it demands efforts from a number of employees;
- errors in test results are possible due to human factor;
- the process may be unstable during the period between tests while the sample collection is quite rare due to restriction of laboratory capacity.

Therefore, there is a need in the art for new simple means to automatically control an iron ore beneficiation in direct iron reduction process and the situation may be generally improved with implementing of automatic rapid measuring test equipment offered in the disclosure. This will provide process control algorithms and will significantly increase the product quality and output stability.

SUMMARY OF THE INVENTION

The object of this invention is to provide means for fast and reliable automatic estimating the quality of the pellets during the direct iron reduction processes based on the density of the metallized iron pellets to ensure stability of the direct iron reduction process and high level of iron output to increase the product quality.

In particular, an object of the present invention is to provide an apparatus for automatic controlling a direct reduction process of an iron oxide containing material, the apparatus comprising

- sampling means adapted to periodically take samples of metallized pellets produced during the reduction process of the iron oxide containing material,
- a mill device for milling the samples of the metallized pellets,
- density determining means to determine a density of the metallized pellet material in the milled sample and
- calculation means adapted to estimate the quality of the reduced metallized

pallets based on the density determined by the density determining means.

The apparatus is adapted to determine a bulk density of the milled metallized pellet material and estimate a metallization of the metallized pellet material as the quality of the metallized pellets based on the bulk density.

5 The apparatus is adapted to control the direct reduction process of the iron oxide containing material by comparing at least two values of the bulk density of the milled metallized pellet material determined by the density determining means or/and by comparing the bulk density of the milled metallized pellet material determined by the density determining means with a predetermined reference bulk density.

10 The predetermined reference bulk density is calculated based on a chemical composition percentage of the metallized pellet material or derived from statistic information on dependency between iron content in the metallized pellets and the bulk density of the milled metallized pellet material in the samples.

15 The sample supply means comprises a supply tube in communication with a metallized pallet flow and with a receiver of the mill device. Preferable, an attritor is used as the mill device.

The density determining means comprises a dosing unit, milled sample transferring means capable to transfer the milled metallized pellet material from the dosing unit to a container having a predetermined volume and a weight unit.

20 The further object of the present invention is to provide a method for controlling a direct reduction process of an iron oxide containing material, the method comprising periodically taking samples of metallized pallets produced during the reduction process of the iron oxide containing material,

25 milling the samples of the metallized pallets to predetermined particle sizes, determining a density of the metallized pellet material in the milled sample and estimating the reduced metallized pallet quality based on the determined density.

Determining the density includes measuring a bulk density of the milled metallized pellet material and a metallization of the metallized pellet material is estimated as the quality of the metallized pellets based on the bulk density.

30 Controlling the direct reduction process of the iron oxide containing material further includes comparing at least two values of the bulk density of the milled metallized pellet material determined by the density determining means or/and

comparing the bulk density of the milled metallized pellet material determined by the density determining means with a predetermined reference bulk density.

The method comprises calculating the predetermined reference bulk density based on a chemical composition percentage of the metallized pellet material or
5 deriving the predetermined reference bulk density from statistic information on dependency between the iron content in the metallized pellets and the bulk density of the milled metallized pellet material in the samples.

The method further comprises adjusting parameters of the direct reduction process when the instability of the periodically determined bulk density is above the
10 predetermined threshold of about 1,5 - 2%. In reduction of iron oxides to metallized iron a bed of iron oxide materials in pellet form is loaded into a shaft furnace with injecting a heated reduction gas, typically a mixture of hydrogen and carbon monoxide, for a sufficient period of time to accomplish substantially complete reduction of the oxides to metallized iron. The proposed solution is based on sampling and weighting of
15 metallized iron pellet output. The sampling operation is automatic and gives test results quite fast. The periodicity of tests may be relatively high up to few minutes and gives information between laboratory tests so the periodicity of laboratory testing could be even decreased.

The proposed test method is based on measurement of weight (m) or density (ρ)
20 stability in the samples. Relatively significant weight or density change of the certain volume (V) of metallized iron pellet sample material indicates loss of process stability, in most cases this leads to decrease of metallized iron material density, i.e. decrease of iron content. Important point is that the volume of the solid material sample should be constant from test to test to obtain accurate determination of density by density
25 calculation from the evident formula

$$\rho = \frac{m}{V},$$

where ρ is density of the milled metallized pellet material,

V is volume and

m is mass of the sample.

30 Particle distribution of pelletized metallized iron material is evidently unstable. There are no means within the process which may precisely control size of metallized iron material pellets. So it is a problem to ensure a certain volume of the sample

material to be weight further. Two ways of treating of pelletizer metallized iron material sample may be proposed. One of them is melting the sample which ensures non-porous homogeneous media which may fill a whole space of some container with certain predetermined volume. The problem of the way is to melt all high-temperature oxides contained in pellets, material waste due to the intensive evaporation and further work with the melt. Another way is to ensure a constant particle distribution in the sample material which means it may fill a certain volume container as a pack of grains with stable porosity and therefore stable solid content. The last way is considered as a main method of sample treatment in the disclosure. Conversion of pelletized metallized iron material into sort of sand may be performed with use of an appropriate mill device, for example an attritor. The equipment may transform sampled material into form of sand or powder with quite stable predetermined particle distribution.

Thus, the present invention provides new simple automatic means for fast and reliable measuring iron ore beneficiation process parameters.

Although this invention will be explained with reference to Midrex process for producing direct reduced iron, the apparatus and the method of the invention may be used in direct iron reduction processes any other types as well as in pelletized ore beneficiation processes or in direct reduction processes for producing other metals.

Fig. 1 illustrates a structure of one exemplary embodiment of the claimed apparatus.

DETAILED DESCRIPTION OF EMBODIMENTS

A structure of one exemplary embodiment of the claimed apparatus for automatic control of the direct iron reduction process is shown Fig. 1. The arrangement of proposed automatic apparatus comprises sampling means adapted to periodically take samples of metallized pellets, for example the sampling means may comprise a supply tube 1. One end of the supply tube 1 is in operable communication with metallized iron pallet flow via a gate 2 and the other end is in communication with a mill device 3. In this embodiment an attritor is preferable used as the mill device. After the gate 2 is open the supply tube 1 feeds the attritor's receiver 4 with beneficiated output pelletized material comprising metallized iron. The attritor 3 mills the sample into form of sand with certain predetermined particle distribution. Recommended average size of normally distributed particles is 0.1-0.4 mm. Constant particle size is acceptable as well

in case this could be provided). After all sand is prepared the dosing unit 5 loads it via an outflow tube 6 into a container 7 with known predetermined volume, preferable blows it with certain low pressure into the outflow tube 6 in communication with the container 7 of known volume. The knife 8 gently removes excess of sand from the top of the container and locks the sample in the certain known volume of container's chamber. The container goes to weighing means, for example weight unit 9 for mass measuring. The weight unit is in communication with calculation means, for example computer with appropriate software which automatically does calculations, in particular calculates a bulk density values for the samples. The calculation means is further adapted to collect the information from all tests online, to estimate the quality of the reduced metallized pellets based on the calculated density values and to inform operator on process deviations and possible problems.

It could be proposed to have 200-300 g of the sand in the milled sample to ensure repeatability of the sand blowing into container, representative pack of sand grains and good sensitivity within mass measurements. The repeatability is very important for accurate density measurements, therefore all conditions of the routine test must be very precisely tuned as constant from test to test including blowing pressure, attritor settings, dosing, work of knife, appropriate cleanness of all instruments (regular maintenance, for example, automatic compressed air cleaning should be performed after every test), etc.

The bulk density of the output metallized pellet material in its milled form may be calculated from known sample mass and the known predetermined container volume and the metallization level of the metallized pellet material may be estimated as the quality of the metallized pellets based on the measured bulk density.

To control the direct reduction process of the iron oxide containing material the claimed invention proposes to compare at least two values of the bulk density of the milled metallized pellet measured on periodically taken samples of the output metallized iron material pellets.

In another embodiment of the invention the bulk density of the milled metallized pellets determined by the density determining means may be compared with the predetermined reference bulk density. As the reference bulk density of the metallized pellets may be used the bulk mix density to be calculated from data concerning

available examples of the chemical composition percentage of the metallized pellet material, for example obtained from previous laboratory tests.

Calculation of a predicted estimated output mix density of the milled metallized pellet material in the bed is based on additive rule:

$$\rho = \frac{(1-k)}{100} \sum_{i=1}^n \rho_i C_i,$$

where

i – number of mix component;

ρ_i – density of the component;

C_i – percentage of the component;

n – total number of components;

k – porosity of the milled pellet material.

The density differences lead to sample mass differences. According to above proposed mass level of sample, applicable level of porosity for average size of the milled pellet sand grains of approximately 0.2mm may be about $k \sim 0.4$ and the container volume should be about 50–100 cm³, preferable about 70 cm³.

Typical composition of pelletized metallized iron output (two versions are presented) are showed in Table.

The Table shows two examples for typical mineralogical/chemical compositions of the output metallized pellet material as well as densities for all components, calculated densities for the solid compound material, calculated densities for the milled granular metallized iron pellet material in the bed and sample mass estimations.

i	Name	Density ρ_i , kg/m ³	Output 1 C_i , %	Output 2 C_i , %
1	Fe	7874	88.88	85.72
2	SiO ₂	2650	1.68	2.12
3	Al ₂ O ₃	3970	0.52	0.75
4	CaO	3320	0.87	1.20
5	MgO	3580	0.46	0.64
6	TiO ₂	4170	0.07	0.10
7	P	1820	0.057	0.057
8	S	2070	0.005	0.004

9	C	2250	1.95	2.50
10	FeO	5870	5.30	6.60
11	MnO	5400	0.20	0.30
12	V ₂ O ₅	3357	0.01	0.01
Total, %			100	100
Calculated compound density (substances 1-12 above), kg/m³			7479	7364
Effective calculated density of the granular metallized pellet material ρ, kg/m³ which is equal to expected measured density			4487	4417
Mass m of the sample container netto ($V=70\text{ cm}^3$), g which is equal to expected measured mass			314.12	309.28

As shown in the table the sample mass is changed for ~5 g if iron output is ~3% weaker, therefore the mass difference value is very significant for high-sensitive weighting device which is used for the tests according to the present invention. Thus, the mass of the milled metallized pellet material in the container of known volume is indicative of process stability. The measured mass values of sample in the container of known volume should be permanent and close to calculated evaluated mass of the sample as shown in Table, wherein the mass difference between the samples should not exceed the predefined range.

Statistic information on dependency between the iron content in the metallized pellets and the bulk density of the milled metallized pellet material in the output flow obtained in previous laboratory tests or bulk density measurements for specific iron ore of iron oxide containing materials can be used also as the predetermined reference bulk density to estimate the quality of the reduced metallized pallets and changes in iron content.

The instability of the periodically determined bulk density between two or more

samples is a significant criterion for establishing deviations in the reduction process. For example, said bulk density of the milled metallized pellets measured in the density determining means according to the invention is compared to at least one or more density values obtained previously and if the difference between said measured bulk density and the previous density value or values is not within a predetermined range, in particular if said difference is equal to or greater than a predetermined percent threshold, the method further comprises adjusting parameters of the direct reduction process of the iron oxide containing material to ensure high level of iron output and the product quality.

Alternatively, said bulk density of the milled metallized pellets measured in the density determining means according to the invention is compared to a predetermined density value that corresponds to appropriate iron level in the output product and if the difference between said measured bulk density and the predetermined reference density is not within a predetermined range, in particular if said difference is equal to or greater than a predetermined percent threshold, the parameters of the direct reduction process may be adjusted.

As mentioned above the predetermined reference density may be derived from previous statistic information regarding the process, in particular from correspondence between iron output and the metallized pellet density, or may be estimated from data regarding chemical composition percentage of the metallized pellet material available from laboratory investigations.

In particular case, if the density instability in the metallized pellet material samples is above the predetermined percent threshold of about 1-3%, preferable of about 1,5 - 2%, the parameters of the reduction process, in particular, gas temperature, gas concentration, amount of gas, oxygen temperature, the optimum bed temperature or other process parameters should be checked and adjusted in order to prevent iron output decreasing.

The apparatus may be additionally configured for outputting an alert when the measured density instability does not meet the mentioned above threshold indicative of process instability to inform operator on process deviations and possible problems.

Some noise factors may affect test accuracy. So it could be recommended to consider the method as a way to indicate iron output instability from the level of about

1-2%. Global increase of accuracy is based on statistic evaluation of test data. The evaluation must be performed initially during adoption of the test method and equipment settings to features of certain facility. Compensation of effect of instability from known uncontrollable (for example, some minor change of material hardness which leads to slight change of attritor output, etc.) and noise factors which affect test accuracy may be used in the form of correction factors determined from statistical analysis using a linear regression algorithm paired with the so-called local-global maps. This algorithm allows estimating the iron pellet (at this stage - sand) quality with high accuracy based on the set of test embedded into it during the calibration phase. It learns the dependencies between the parameters during the calibration period and then is used to calibrate the rough estimations received at the physical test stage.

Both hardware and software can be a product which is capable of estimating the metallization of the iron pellets and can be used in the automation loop as a quality controller. This will allow such productions to operate in a semi-automatic way and be able to do the control actions of the plant nearly online, which is currently not possible due to the laboratory induced delay.

The invention allows increasing direct reduction process stability and improves process control. In particular, the claimed apparatus and method provides following advantages:

- 20 frequent rapid check of material output with relatively simple automatic test;
- number of laboratory tests may be decreased significantly which is economically reasonable as well;
- automaticity of all testing stages ensures absence of errors due to human factor;
- iron oxide containing material or ore material consistency and production
- 25 process stability are improved;
- online control of the production process;
- creation of the device for the quality control loops and process parameters optimization.

It should be noted also that the proposed method is quite universal for any iron oxide containing materials or iron ore with known particle shapes and may be used also for input control of iron oxide containing material or iron ore as well. This method may be used also in any desired step of the reduction process. Further, the proposed method

may be adapted for input or output control in direct iron reduction processes of any other types or in pelletized ore beneficiation processes and in direct reduction processes for producing other metals.

5 Although this invention has been described in detail with reference to certain preferred embodiments, it should be appreciated that the present invention is not limited to those precise embodiments. The disclosure above describes the best embodiments of the invention and many modifications and variations are possible without departing from the scope and spirit of this invention and lie also within the scope of the present invention.

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CLAIMS

1. An apparatus for automatic controlling a direct reduction process of an iron oxide containing material, the apparatus comprising
sampling means adapted to periodically take samples of metallized pellets
5 produced during the reduction process of the iron oxide containing material,
a mill device for milling the samples of the metallized pellets,
density determining means to determine a density of the metallized pellet material in the milled sample and
calculation means adapted to estimate the quality of the reduced metallized
10 pellets based on the density determined by the density determining means.
2. The apparatus according to claim 1, wherein the apparatus is adapted to determine a bulk density of the milled metallized pellet material and estimate a metallization of the metallized pellet material as the quality of the metallized pellets based on the bulk density.
- 15 3. The apparatus according to claim 1 or 2, wherein the apparatus is adapted to control the direct reduction process of the iron oxide containing material by comparing at least two values of the bulk density of the milled metallized pellet material determined by the density determining means or/and by comparing the bulk density of the milled metallized pellet material determined by the density determining means with
20 a predetermined reference bulk density.
4. The apparatus according to claim 3, wherein the predetermined reference bulk density is calculated based on a chemical composition percentage of the metallized pellet material.
5. The apparatus according to claim 3, wherein the predetermined reference bulk
25 density is determined from statistic information on dependency between iron content in the metallized pellets and the bulk density of the milled metallized pellet material in the samples.
6. The apparatus according to claim 1, wherein the sample supply means comprises a supply tube in communication with a metallized pellet flow and with a
30 receiver of the mill device.
7. The apparatus according to claim 1, wherein the mill device is an attritor.
8. The apparatus according to claim 1, wherein the density determining means

comprises a dosing unit, milled sample transferring means capable to transfer the milled metallized pellet material from the dosing unit to a container having a predetermined volume and a weight unit.

5 9. A method for controlling a direct reduction process of an iron oxide containing material, the method comprising

periodically taking samples of metallized pellets produced during the reduction process of the iron oxide containing material,

10 milling the samples of the metallized pellets to predetermined particle sizes, determining a density of the metallized pellet material in the milled sample and estimating the reduced metallized pellet quality based on the determined density.

10. The method according to claim 9, wherein determining the density includes measuring a bulk density of the milled metallized pellet material and a metallization of the metallized pellet material is estimated as the quality of the metallized pellets based on the bulk density.

15 11. The method according to claim 10, wherein controlling the direct reduction process of the iron oxide containing material further includes comparing at least two values of the bulk density of the milled metallized pellet material determined by the density determining means or/and comparing the bulk density of the milled metallized pellet material determined by the density determining means with a predetermined
20 reference bulk density.

12. The method according to claim 11, comprising calculating the predetermined reference bulk density based on a chemical composition percentage of the metallized pellet material.

25 13. The method according to claim 11, comprising determining the predetermined reference bulk density from statistic information on dependency between the iron content in the metallized pellets and the bulk density of the milled metallized pellet material in the samples.

30 14. The method according to any one of claims 9 to 13 further comprising adjusting parameters of the direct reduction process when the instability of the periodically determined bulk density is above the predetermined threshold of about 1,5 - 2%.

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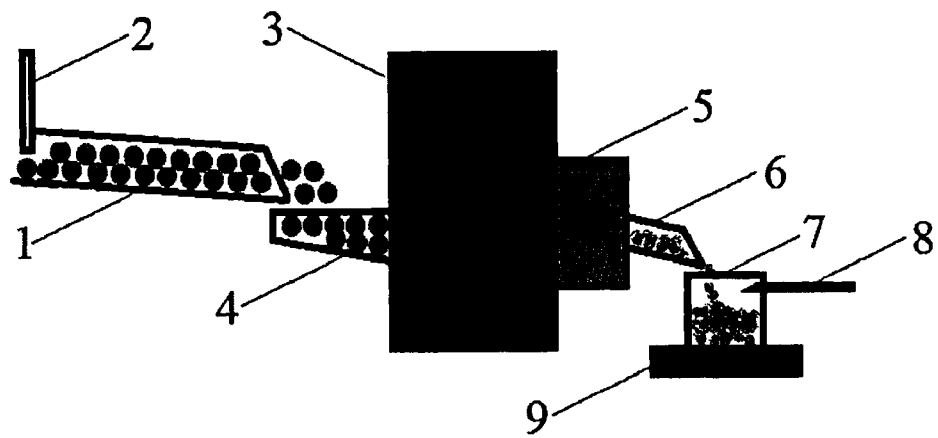


Figure 1

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER

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ADD.

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B. FIELDS SEARCHED

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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.
X	CA 2 266 301 A1 (KOBE STEEL LTD [JP]) 24 September 1999 (1999-09-24)	1,3,4, 6-10,12, 13
A	figure 8 abstract	2,5,11, 14
X	WO 2012/002338 A1 (KOBE SEIKO SHO KK) 5 January 2012 (2012-01-05)	1,3,4, 6-10,12, 13
A	abstract table 1	2,5,11, 14
E	US 2013/081516 A1 (SIMMONS JOHN J [US]) 4 April 2013 (2013-04-04) paragraph [0077]	1,9



Further documents are listed in the continuation of Box C.



See patent family annex.

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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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