This invention relates to a process for the reduction of complex ores whereby the metallic components of the ore can be recovered and substantially separated. It relates more particularly to an iron ore having associated therewith reducible compounds of other metals as for example, oxides, some of which other metals it may be desired to alloy with a part of the iron, while others it is desired to separate from the first alloy, or to form a separate alloy with the balance of the reducible iron.

An object of this invention is to produce a metallic product suitable for furnace treatment, which is an alloy of the major metal, as for example iron, with a secondary metal, as for example nickel.

Another object of this invention is to effect the reduction in a subsequent operation of further metallic components of the ore, remaining unreduced after a first reduction process, for the purpose of producing a metallic product suitable for furnace treatment, which is an alloy of said unreduced components of the ore.

A particular ore which is amenable to treatment by my process is the Mayari iron ore of Cuba, said ore being mainly a limonite associated with chromite, and containing approximately 2.00% chromium, 75% nickel and 0.8% manganese. This ore also contains a high percentage of moisture, which on removal leaves a product, mainly in a state of fine subdivision, unsuitable for a blast furnace charge. An agglomerating process is therefore necessary for the purpose of rendering the moisture content and at the same time the ore in a suitable condition to be charged to the blast furnace. The blast furnace product is a chromiferous pig iron, containing approximately 2% of chromium and 1% of nickel. The chromium may be successfully removed from this chromiferous pig iron by Bessemerizing processes known to the art and the decromized iron containing nickel may be subsequently treated in the open hearth or in the electric furnace in the well known manner.

It is, however, a purpose of this invention to produce a metallic iron product direct from the ore, having associated with it substantially all of the nickel originally contained in the ore, but practically free of chromium, and which will be suitable for charging directly into refining metallurgical furnaces, as for example, an electric furnace. In this manner I intend to eliminate the operations of agglomerating, of blast furnace reduction, and of Bessemerizing, hitherto practiced in the manufacture of steel from this ore. The retention of the nickel with the primary metallic product obtained by my process is obviously advantageous for the manufacture of nickel steels.

In this method of reduction I take advantage of the relative disposition to reduction of the various metallic components of the ore. It is well known that nickel is reduced more readily than iron, and that iron is reduced more readily than chromium at appropriate temperatures, in the presence of a reducing agent, as for example, carbon, carbonaceous gases or hydrogen. In other words, a reducing agent such as carbon, will take its oxygen from a nickel oxide in preference to taking it from a chromium oxide. By applying this principle of preferential reduction by means of reducing agents, such as carbon, carbonaceous gases or hydrogen, I have found that the extent of the reduction may be controlled, as desired, within substantially narrow limits. This may be effected by employing a limited amount of the reducing agent sufficient only to reduce the oxides of metals, or such portions of them, as it may be desired to reduce, provided said metals are those in the lower order of sequence with respect to their relative disposition to reduction, that is to say, in the example I have given, the order of sequence would be: nickel, iron, chromium.

I have found that an addition of coal amounting to 15% of the ore charge, said addition having been intimately mixed with the ore, and submitted to a temperature of approximately 1800° F., results in the reduction of substantially all the nickel and about 70% of the iron, without however effecting a reduction of the manganese and silicon, and only very slightly reducing the chromium.

With an addition of 25% coal, and suitable temperatures as above described, a greater amount of the iron is reduced and a considerable portion of the chromium is also reduced, the latter to the extent of approximately 30% of that present in the ore. With increasing quantities of coal the reduced metallic portion will carry further additional amounts of chromium.

In carrying out my invention I mix fine coal
or other carbonaceous material with the finely-divided ore, the coal in one instance being 15% of the weight of the ore. This mixture is charged to a suitable furnace, as for example, a rotary kiln furnace, and submitted therein to the direct heat of combustion gases. At a temperature of 1800°F, I have found that the nickel in the ore is entirely reduced and also about 70% of the iron, but the chromium sesquioxide, the silica, alumina, magnesia and oxide of manganese present, are practically unaffected. By increasing the percentage of coal incorporated with the ore and employing a suitable temperature, I may, however, also effect a substantial reduction of the chromium, as shown in the appended table.

<table>
<thead>
<tr>
<th>Coal added</th>
<th>Ni content</th>
<th>Fe content</th>
<th>Cr content</th>
<th>Per cent recovery</th>
</tr>
</thead>
<tbody>
<tr>
<td>percent of ore charged</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Original ore</td>
<td>88</td>
<td>98.8</td>
<td>99.75</td>
<td>99.8</td>
</tr>
<tr>
<td>Reduction product</td>
<td>22%</td>
<td>2.31</td>
<td>99.9</td>
<td>95.36</td>
</tr>
<tr>
<td>Reduction product</td>
<td>25%</td>
<td>1.95</td>
<td>99.8</td>
<td>95.36</td>
</tr>
</tbody>
</table>

My present object however is to confine the primary reduction and metallizing of the ore to the recovery of nickel and iron, in suitable proportions to form a nickel iron alloy substantially free from chromium and to effect the reduction of the chromium in a second operation.

In order to subsequently reduce the chromium, it is necessary to separate the products of the primary reduction, so that the reduced portion may be utilized in a refining furnace for the manufacture of a nickel steel, while the unreduced portion may be submitted to a secondary process of reduction for the recovery of the balance of the iron with the major part of the chromium, so as to form a chromium iron alloy.

It will be obvious to those skilled in the art, that the combined products of my primary reduction process may be heated to a temperature corresponding to the fusion points of the reduced metals, which in the example I have given will be principally nickel and iron. At these temperatures the metallized particles may be caused to flow from the slag, in a manner well known in metallurgical practice.

It will also be obvious that the heating of the metallized particles to their fusion point may be carried out, either in the same furnace as that which has been used for reduction, or in a separate furnace as a subsequent operation. The process is likewise susceptible of further modification, so as to complete, in said subsequent melting operation, the partial reduction previously effected in the reduction furnace. Furthermore, said melting operation may be associated with such refining steps as may be required for the production of a finished steel product. The slag remaining may then be submitted to a further process of reduction, in order to metallize the unreduced chromium and/or other desired metallic components present therein.

The separation of the metalized particles may however be effected in a somewhat different manner. I have discovered that by raising the temperature of the product, resulting from the primary reduction, to a point between 2300° and 2400°F, the reduced or metallized portions dispersed in the mass are caused to amalgamate, while the slag forming portion remains unchanged. On cooling and crushing the mass, this metallic amalgamation is readily separable from the slag by magnetic, by electro-static or by mechanical means, and is then in a condition eminently suitable for a refining operation in a metallurgical furnace. The slag portion may be further treated for the recovery therefrom of chromium, and/or other desired metals, by the action of a reducing agent, such as carbon, in amount sufficient to reduce these metallic components of the slag, said reduction being effected in a manner similar to that already described for the reduction of the nickel and of the major portion of the iron. This secondary reduction results in a metallized product comprising metallic chromium, the balance of the iron left unreduced in the first reduction process, and small amounts of manganese and silicon; the remaining constituents of the ore forming the secondary reduction slag.

While I have taken as an example of my process a nickel bearing chromiferous iron ore, it is evident that the process is equally applicable to any complex ore containing reducible metallic compounds, and that a similar selective reduction of any of said compounds may be effected in the manner aforementioned, provided the desired selection is in the natural sequence of their individual dispositions to reduction. It is also evident that one of the means employed in the second step of my process, devised to effect the amalgamation of metallic particles widely diffused in a complex mass, (said amalgamation being a preliminary step to their separation from the slag by magnetic, electro-static or other mechanical means), is susceptible of wide application. Hence it will be obvious to those skilled in the art that my invention is susceptible of various changes and modifications without departing from the spirit thereof, and I desire, therefore, that only such limitations shall be placed thereupon as are imposed by the prior art, or as are specifically set forth in the appended claims.

In certain of the claims which follow, I refer to my process being applicable to the separation of components not widely different in their reduction temperatures. By this I intend to convey the idea that the process is applicable to complex ores or other associa-
tions of metal compounds in which the different metal compounds will be reduced together unless special care is taken. Obviously, I do not mean that the temperatures of reduction are approximately the same. The specific embodiment of the invention which has been used to illustrate it clearly indicates that this is not my meaning. In this specific example the reduction temperatures of nickel and chromium are appreciably different, and yet they would be reduced together unless special care were taken to prevent this occurring. What I wish to distinguish from are those ores in which the components are so widely separated in their temperatures of reduction that there is relatively little danger of reducing one compound when reducing the other.

Having thus described my invention what I claim as new and desire to secure by Letters Patent is:

1. The process of selectively reducing the reducible components of a complex ore which components are not widely different in their reduction temperatures, which consists in, heating the ore to a suitable temperature with sufficient reducing agents to substantially reduce such of the more readily reducible components it may be desired to separate primarily from the ore, said reducing temperature being lower than the fusion point of the desired component, in heating the mass after said reduction has been effected to a higher temperature at which a substantial agglomeration of the dispersed metallic particles may be effected, in cooling and in crushing the mass, in effecting the mechanical separation of the metallic agglomerate from the unreduced portion of the mass, and in submitting said unreduced portion to a secondary reduction by heating it at a suitable temperature with sufficient reducing agent to reduce such further components as may be desired.

2. The process of selectively reducing a nickel bearing chromiferous iron ore, which consists in heating the ore to a temperature preferably ranging from 1750 to 1900°F. in the presence of a carbonaceous reducing agent in amount substantially that required to reduce the nickel and a major portion of the iron present in the ore so as to effect their reduction, in raising the temperature of the mass, after said reduction has been effected, to a temperature preferably ranging from 2400°F. to 2500°F. so as to agglomerate the reduced metal, in cooling and in crushing the mass, in separating the agglomerate from the slag and in submitting said slag to further reduction in the presence of sufficient carbonaceous reducing agent to reduce the chromium and other reducible metallic compounds present in the slag.

3. The process of selectively reducing a nickel bearing chromiferous iron ore, which consists in intimately mixing with the ore substantially sufficient carbonaceous reducing agent to reduce the oxides of nickel and parts of the oxides of iron present in the ore, in roasting the mixture in a suitable furnace at a temperature preferably between 1750°F. and 1900°F. so as to effect a reduction of the nickel and of the major part of the iron, in further heating the roasted mass to a temperature preferably between 2400°F. and 2500°F. so as to agglomerate the metallic particles dispersed therein, in cooling and crushing the mass, in separating the crushed metallic agglomerate from the unreduced portion of the mass by magnetic separation, and in submitting said unreduced portion to a further reduction by roasting it at a suitable temperature in the presence of substantially sufficient carbonaceous reducing agent to reduce the oxide of chromium and other reducible compounds present in said unreduced portion.

4. The process of selectively reducing a nickel bearing chromiferous iron ore, which consists in intimately mixing with the ore substantially sufficient carbonaceous reducing agent to reduce the oxides of nickel and part of the oxides of iron present in the ore, in roasting the mixture in a suitable furnace at a temperature preferably between 1750°F. and 1900°F. so as to effect the reduction of the nickel and of the iron, in further heating the roasted mass to a temperature preferably between 2400°F. and 2500°F. so as to agglomerate the metallic particles dispersed therein, in cooling and crushing the mass, in removing the crushed metallic agglomerate from the unreduced portion of the mass by mechanical separation, and in submitting said unreduced portion to a further reduction by roasting it at a suitable temperature in the presence of substantially sufficient carbonaceous reducing agent to reduce the oxide of chromium and other reducible compounds present in said unreduced portion.

5. The process of selectively reducing a nickel bearing chromiferous iron ore, which consists in intimately mixing with the ore substantially sufficient reducing agent to reduce the oxides of nickel and part of the oxides of iron present in the ore, in roasting the mixture in a suitable furnace at a temperature preferably between 1750°F. and 1900°F. so as to effect the reduction of the nickel and of the iron, in further heating the roasted mass to a temperature preferably between 2400°F. and 2500°F. so as to agglomerate the metallic particles dispersed therein, in cooling and crushing the mass, in removing the crushed metallic agglomerate from the unreduced portion of the mass by any suitable separating process, and in submitting said unreduced portion to a further reduction by roasting it at a suitable temperature in the presence of substantially sufficient re-
Reducing agent to reduce the oxide of chromium and other reducible compounds present in said unreduced portion.

6. The process of selectively separating the reducible components of a complex mass which components are not widely different in their reduction temperatures, which consists in submitting the mass to serial reductions, each of said reductions being effected under the influence of sufficient reducing agent and at temperatures suitable for reducing principally a desired component, said temperatures being in each case lower than the fusion point of the desired reduction product, and raising the temperature at the end of each reduction to a degree favorable to agglomeration of the reduced components, but insufficient to fuse the mass, and separating the agglomerate from the remainder of the mass.

In testimony whereof I hereunto affix my signature this twenty first day of November, 1924.

FRANK O. KICHLINE.

CERTIFICATE OF CORRECTION.

Patent No. 1,717,160. Granted June 11, 1929, to

FRANK O. KICHLINE.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 1, line 9, for the word "is" read "may be"; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 13th day of August, A. D. 1929.

M. J. Moore,
Acting Commissioner of Patents.