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REFINING NICKEL

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5 Claims. (Cl. 75—82)

This invention relates to metallurgy and more particularly to nickel metallurgy and has for its object the provision of a method of producing substantially pure nickel from finely divided or comminuted nickel containing relatively small amounts of residual impurities.

Another object is to facilitate the production of substantially pure nickel.

Still another object is to provide a method of processing nickel metal powder to eliminate therefrom substantially all residual impurities to obtain thereby a high grade substantially pure nickel metal powder product.

Other objects will be apparent as the invention is more fully hereinafter disclosed.

In accordance with these objects I have discovered that when impure metallic nickel in its finely divided form is mixed with nickel chloride ($NiCl_2$) and the mixture is heated to elevated temperatures within the range 500 to 1000° C. for an extended time interval, the metallic impurities present in the metallic nickel are in major part converted into metal chloride compounds by a displacement reaction between the contaminating metal and the nickel chloride, the extent of such conversion being dependent upon metal particle size, the amount and form of said impurities, the temperature and time of heating and other factors.

The reaction involved in this invention is essentially one of displacement wherein the metallic impurity displaces the nickel of the nickel chloride, the metal and chloride compounds each being in the heated but substantially solid phase.

Contrary to expectations based on all of the data heretofore published, I have discovered that nickel is relatively low in the displacement series in the system metal-metal chlorides at temperatures within the range 500–1000° C., and that most of the metals commonly associated with metallic nickel as difficultly removed impurities, such as Cu, Co, Fe, Cr, Zn and Mn, lie above nickel in the said series and will displace nickel from nickel chloride at some temperature within this range.

The melting point of nickel chloride has not been accurately determined. The melting point approximates 1001° C. and the boiling point approximates 975° C. When heated alone, nickel chloride appears to vaporize just before melting and seems to have a melting point in the neighborhood of 1000° C.

Irrespective of this peculiarity of nickel chloride, I have found that when nickel chloride in its anhydrous finely divided solid form is heated to temperatures as low as 500° C. and as high as 1000° C. in the presence of any one of the metals Cu, Co, Fe, Cr, Zn and Mn, the nickel content of the nickel chloride is progressively thrown out of chloride combination with formation of the chloride compound of the metal in contact therewith, the progress of the reaction being one based on the relative rates of diffusion of the respective metals into each other and the rates of diffusion of the chlorides into each other, at any given temperature of heating.

Therefore, by finely comminuting both the metal and the nickel chloride, to reduce the diffusion paths to the smallest economically practical distance and briquetting the mived metal and chlorides to bring the particles into relatively close contact, the time factor may be lowered correspondingly and by employing a relatively large excess of nickel chloride over that theoretically required the rate of the reaction at any given temperature may be accelerated.

In the adaptation of this invention commercially, the most practical application of the same appears to be in the treatment of nickel metal powder to eliminate therefrom from small percentages down to substantially residual percentages of the metallic impurities Cu, Co, Fe, Cr, Zn and Mn, present therein thereby to obtain a nickel metal powder for sintering heat-treatment to solid or coherent form that is of a relatively high degree of purity.

Heretofore in the art the preparation of substantially pure nickel has depended upon the practice of complex and expensive purification processes prior to reduction of the nickel compound to finely divided metallic form. Even by the practice of these complicated and expensive chemical processes it is difficult to obtain substantially pure nickel. In accordance with the practice of the present invention, however, the major portion of the prior art complex and expensive chemical processes may be dispensed with and a reduced metal product consisting mainly of nickel and containing up to 2% metallic impurities consisting of any one or more of the metals Cu, Co, Fe, Cr, Zn and Mn, may be produced which on comminuting to relatively small particle size, mixing with nickel chloride, briquetting and heat-treating to temperatures within the range 500 to 1000° C., will be converted into substantially pure nickel.

As one specific embodiment of the present invention, but not as a limitation thereof, the following example may be given:

Most nickel sulfide ores contain copper in var-

ious amounts, the complete separation of which from the nickel is difficult. Two main processes are at present in use, the Orford process based on the relative solubility of Cu and Ni sulfides in sodium sulfide and the Mond process based on the volatility of nickel carbonyl. Each of these processes are complicated and costly.

In accordance to the practice of the present invention the nickel and copper content of the ore are isolated as metal oxides by methods heretofore known in the art and the mixed metal oxides are reduced to metal powder by reduction in hydrogen, in accordance with well known prior art practice, the reduction temperature preferably being maintained as low as possible to obtain as small a particle size as practicable to handle subsequently, in accordance with the present invention. Higher reduction temperatures may be employed, however, with no essential departure from the present invention as one skilled in the art will recognize.

The metal powder product obtained as above noted is screened to pass about 100 mesh screen, the over sized particles being rejected or crushed and screened to pass 100 mesh, and is mixed with nickel chloride (anhydrous) also crushed to pass about 100 mesh, the relative proportions of metal powder to nickel chloride being such as to provide from 2 to 5 times the amount of nickel chloride theoretically required to obtain the substantially complete conversion of the copper content of the metal powder to copper chloride.

This mixture is then compacted to suitable sized aggregates or briquets and the briquets are heated to temperatures approximating 700° C. for an extended time interval approximating one hour, in an inert or substantially non-oxidizing atmosphere, such as pure dry nitrogen, and the resulting sintered product is treated for the separation of the metal powder from the mixed cuprous and nickel chlorides present therein.

The reaction involved is essentially as follows:

$$NiCl_2+2Cu=Ni+2CuCl$$

The cuprous chloride reaction product has a melting point approximating 425° C., but probably forms a lower melting eutectic with the nickel chloride at temperatures approximating 700° C. In the presence of large excesses of $NiCl_2$ such lower melting eutectics, however, are substantially localized to the surface of the metal particles.

The above heating may be practiced as a batch operation or as a continuous operation, as one skilled in the art will perceive.

I have found that the heat-treatment of nickel metal powder containing up to 2% copper, in the manner described above, is effective to convert substantially all of the copper to cuprous chloride within a time interval of about one hour, using about 3 times the amount of $NiCl_2$ theoretically required to accomplish this result. However, in the case of copper, the subsequent separation of the cuprous chloride from the purified nickel metal powder is rendered difficult by reason of the fact that in aqueous chloride solutions nickel lies above copper in the displacement series. Accordingly, following sintering and conversion of the copper content of the metal powder to cuprous chloride (CuCl) the separation of the nickel metal powder from the mixed copper and nickel chlorides by aqueous solutioning methods must be practiced with the shortest possible time interval of exposure of the metal powder to the displacement reaction ($2CuCl+Ni=NiCl_2+Cu$) which normally occurs in aqueous solution.

Alternatively, by feeding the sinter after cooling to atmospheric temperatures into a molten bath consisting of a mixture of sodium and potassium chlorides the major portion of the copper chloride can be dissolved out of the sinter and decanted away from the metal powder before subjecting the nickel powder to washing with water.

Substantially the same procedure may be followed in separating from the nickel metal powder any of the metals Fe, Co, Cr, Zn and Mn. Iron and cobalt in amounts as high as 2% to 3% may be converted to chloride compounds by the sintering heat-treatment of nickel metal powder contaminated with these metals with nickel chloride in excess of the amount theoretically required at temperatures approximating 700° C. in a substantially non-oxidizing and non-reducing atmosphere.

In practicing the present invention with solid coherent metal, the metal, if not of thin section, is first reduced to small particle size by any convenient mechanical means. A particle size passing 100 mesh has been found to be most suitable for the purposes of the present invention. Thin sectioned metal, such as turnings for example, usually need to be broken up into small sized fragments having a size that is convenient to handle.

It is believed apparent from the above description that the present invention may be widely varied without essential departure therefrom and all modifications and departures therefrom are contemplated as may fall within the scope of the following claims.

What I claim is:

1. The method of treating impure nickel to remove therefrom associated metallic impurities which comprises comminuting the nickel to relatively small particle size, mixing therewith anhydrous nickel chloride in an amount in relatively large excess of the amount required to react with said associated impurities, compacting the mixture and heating the said compacted mixture in an inert and substantially non-oxidizing atmosphere to elevated temperatures within the range 500 to 1000° C. for an extended time interval adapted to convert the major portion of the said associated metal impurities into metal chloride compounds by a displacement reaction with said nickel chloride, and separating the resulting metal product from the chloride compounds associated therewith.

2. The method of claim 1, wherein the product of said heating is immersed in a molten bath consisting of highly stable metal chloride compounds to separate the major portion of the metal chloride compounds therefrom, and wherein the metal after separating the same from the major portion of the said molten bath is washed with water to remove the remainder of the metal chlorides present therein.

3. The method of producing substantially pure nickel which comprises forming an impure nickel metal powder of relatively small particle size, mixing the said powder with anhydrous nickel chloride, the amount of chloride being in large excess of the amount theoretically required to convert all of the metal impurities of the said powder to chloride compounds, compacting the mixture to small sized aggregates, heating the said aggregates in an inert and substantially non-oxidizing atmosphere to a temperature approximating 700° C. for an extended time interval adapted to obtain a conversion of the major portion of said metal impurities to chloride compounds, cooling the sintered aggregates and sepa-

rating the metal powder product remaining from the metal chloride compounds.

4. The method of claim 3, wherein the last mentioned step consists in immersing the sintered aggregates in a molten bath consisting of sodium and potassium chlorides to dissolve out the major portion of said metal chloride compounds and wherein, after separating the major portion of the molten chloride bath from the said metal powder, the remaining chlorides are dissolved in water.

5. The method of treating comminuted nickel to extract therefrom any of the metals Cu, Co, Fe, Cr, Zn and Mn, associated therewith in relatively small total percentages, which comprises mixing the comminuted nickel with anhydrous nickel chloride, the amount of said chloride being from 2 to 5 times that theoretically required to convert the said associated metals into chloride compounds, compacting the mixture into convenient sized briquets, heating the briquets in an inert atmosphere to a temperature approximating 700° C. for an extended itme interval adapted to obtain a substantially complete conversion of the said associated metals to chloride compounds, cooling the heated briquets and separating the metal product remaining from the chloride compounds associated therewith.

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