

The changing landscape of carbonyl iron and nickel powder production

Until recently, much of the global production of carbonyl metal powder was centred on manufacturers in Europe and North America. Although China has produced these powders since the 1960s, the last ten years or so has seen a rapid increase in the volume of both carbonyl nickel and carbonyl iron powders produced in this region. In this report, Jun Shu and Lou Koehler describe the production processes and look at the changing landscape of carbonyl metal powder production.

Carbonyl nickel refining was first commercialised in 1902 by the Mond Nickel Company Ltd, with carbonyl iron powder production being developed in 1925 by Germany's BASF (formerly I G Farben). Today, carbonyl nickel powders are widely used in Powder Metallurgy, battery and fuel cell electrodes, hardmetal binders, welding rods, high-temperature filters, conducting additives, electronic materials, anti-seize lubricants, chemicals and catalysts (Fig. 1). Typical applications of carbonyl iron powders include Metal Injection Moulding (MIM), magnetic cores, hardmetal binders, radar absorption materials, magneto-rheological fluids for shock/vibration damping, precision polishing, industrial diamond synthesis and health supplements.

Global carbonyl nickel powder production capacity has reached around 37,000 metric tons per year, with the main producers being Vale in Canada and Wales, China's Jinchuan and Norilsk in Russia. Carbonyl

iron powder production capacity is reported to be around 29,000 metric tons per year, with BASF being the largest manufacturer. However, nearly half of the current carbonyl iron powder capacity is represented by recent additions from China.

Carbonyls of nickel and iron, of molecular forms $\text{Ni}(\text{CO})_4$ and $\text{Fe}(\text{CO})_5$,

were discovered by Dr Ludwig Mond and his colleagues in 1890 and 1891, respectively [1, 2]. Subsequently, carbonyl nickel refining, or the Mond process, was first commercialised in 1902 by the Mond Nickel Company in Clydach, South Wales, to produce high-purity nickel pellets [3]. Carbonyl nickel powder



Fig. 1 Commercial applications of discrete carbonyl nickel powder

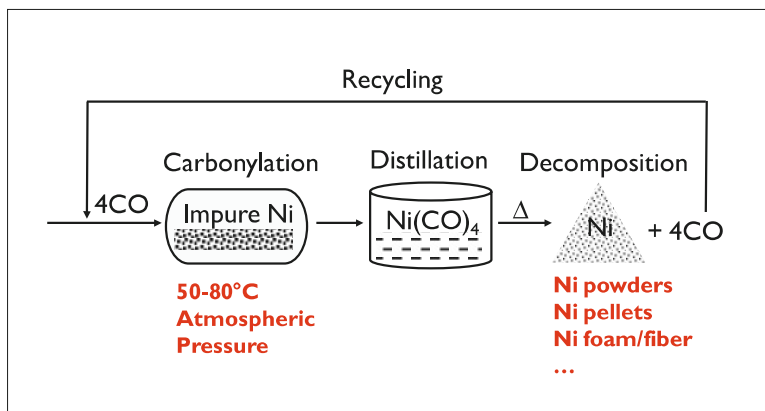


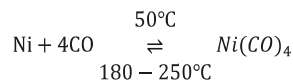
Fig. 2 Schematic diagram of carbonyl nickel extractive metallurgy

production was an invention of the German company BASF in the late 1920s with nickel matte feedstock acquired from the Mond Nickel Company [4].

The International Nickel Company (Inco) acquired the Mond Nickel plant in the 1920s and, in 1943, commercial nickel powder production began. In 1973, Inco opened a second carbonyl plant in Sudbury, Canada, the site of its major nickel mining operations [5]. These plants have been continually modernised and now operate under the ownership of the Brazilian mining giant Vale SA. This mature process is acknowledged as the best available technology for refining pure nickel. The three main reasons for this are the ability to produce a very high purity product, the low energy levels consumed in the process and the fact that, although the process uses highly toxic process intermediates, there are no polluting waste products, as virtually all the carbon monoxide gas is recycled and emissions of toxic carbonyls are controlled in the parts-per-billion concentration range.

The original carbonyl process, still used at Vale's Wales refinery, harnesses the ability of nickel in an impure form to be extracted into a nickel carbonyl gas (boiling point 43°C) at ordinary temperatures and then restored to a pure metallic state by gentle heating. Production begins with a nickel oxide feedstock. The nickel oxide is continuously

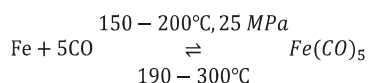
fed into a reduction kiln, where it is tumbled in a stream of pure hydrogen at ~230°C to produce impure nickel in granular form. In the second stage, the volatilisation kiln, the nickel reacts with carbon monoxide at close to atmospheric pressure to form nickel carbonyl gas, or nickel tetracarbonyl Ni(CO)₄:



$$K_{\text{eq}} (50^\circ\text{C}) = 3.78 \times 10^4 \quad (1)$$

The nickel carbonyl gas is then piped to an adjacent plant for thermal decomposition into pure nickel pellets or powders. To produce powder, the nickel carbonyl gas is injected at a metered rate into the top of the decomposer towers. The walls of the towers are heated to 300-500°C. The gas decomposes instantly to form nickel powder which settles at the bottom of the unit. The powder is collected, blended for uniformity, screened and packaged. A schematic of the carbonyl nickel extractive metallurgy is shown in Fig. 2.

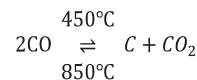
When an iron component is present in the feedstock, iron carbonylation occurs to form iron carbonyl, or iron pentacarbonyl Fe(CO)₅:



$$K_{\text{eq}} (50^\circ\text{C}) = 5.67 \times 10^{-2} \quad (2)$$

At ambient pressure, as practised at Vale's Wales refinery, the iron carbonyl formation equilibrium is negligible in comparison with nickel carbonyl formation. However, this parallel reaction equilibrium increases with pressure, which occurs at the more modern facilities in Vale Canada, Norilsk and Jinchuan, thus making it necessary to separate nickel carbonyl from iron carbonyl in a high pressure carbonyl process in order to produce pure nickel products. In fact, high pressure is fundamental to carbonyl iron synthesis in order to produce carbonyl iron powders upon thermal decomposition.

With the increase of decomposition temperature, a side reaction of CO disproportionation, or the Boudouard reaction, becomes important, resulting in residual carbon deposition on nickel and/or iron particles in specific refining processes:



$$K_{\text{eq}} (250^\circ\text{C}) = 9.53 \times 10^7 \quad (3)$$

Although the Boudouard reaction is thermodynamically favourable at low temperature, its reaction rate is kinetically low until about 450°C in the atmospheric pressure decomposition process. Increasing pressure shifts the position of equilibrium towards the right-hand side, resulting in more residual carbon formation.

The beauty of carbonyl metal refining (for nickel or iron) is that, following the metal extraction and distillation, gaseous metal carbonyl can be thermally decomposed back to high purity metal and carbon monoxide. Under precisely controlled thermal decomposition conditions such as temperature, feed rate, carbonyl concentration, partial pressure and introduction of additives, carbonyl nickel, iron or ferronickel powders can be produced in different morphologies and particle size distributions ranging from micron size powders to centimetre size pellets.

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