PURIFICATION OF METALS

Mond Process

Some metals are purified by obtaining their volatile carbonyl compounds which on heating strongly decompose to yield pure m'etal. Purification of nickel is done by this method. Impure nickel is reacted with carbon monoxide at 325 K to give volatile nickel carbonyl leaving solid impurities behind. Pure nickel is obtained by heating nickel carbonyl at 450-475 K:

$$Ni(s) + 4CO(g) \xrightarrow{325 \text{ K}} Ni(CO)_4(g) \xrightarrow{450-475 \text{ K}} Ni(s) + 4CO(g)$$

Van Arkel de Boer Process

This method is based on the thermal decomposition of a volatile metal compound like an iodide. In this method, firsta metal iodide is formed by direct reaction of iodine and the metal to be purified at a temperature of 475-675 K in an evacuated'vessel. The vapouks of metal iodide, thus formed are heated strongly on a tungsten or tantalum filament at 1300-1000 K. The metal iodide decomposes to yield the pure metal, as in the case of zirconium.

$$Zr(s) + 2I_2(g) \xrightarrow{473-673 \text{ K}} ZrI_4(g) \xrightarrow{1300-1800 \text{ K}} Zr(s) + 2I_2(g)$$

Titanium is also purified by this method. The impure metal is heated with iodine and Ti14 thus formed is decomposed by heating at 1700 K over tungsten filament:

$$Ti(s) + 2I_2(g) \longrightarrow TiI_4(g) \frac{1700 \text{ K}}{\text{Tungsten filament}} > Ti(s) + 2I_2(g)$$

The regenerated iodine is used over and over again. This process is very expensive and is employed for the preparation of limited amounts of very pure metals for special uses.

Zone Refining

This method is used to obtain metals of very high purity. The basic principle involved in this process is similar to fractional crystallisation. A small heater is used to heat a bar of the impure metal. The heater melts a small band of metal as it is slowly moved along the rod. As small bands of metal are thus melted sequentially, the pure metal crystallises out of the melt, while impurities pass into the adjacent molten zone. The impurities thus collect at the end of the bar. This end can be cut off and removed. High grade germanium and silicon are obtained by purifying them by zone refining (Fig. 1).

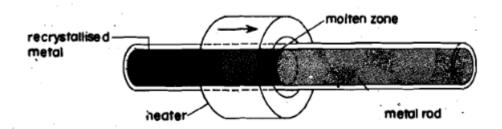


Fig. 1 : Diagram for zone refining

Gold parting is the separating of gold from silver. Gold and silver are often extracted from the same ores and are chemically similar and therefore hard to separate. Over the centuries special means of separation have been invented.

The very earliest precious metals had mixes of gold and silver; gold and silver alloy is called electrum. With the advent of coinage, methods had to be invented to remove impurities from the gold so that gold of specific purities could be made. Cupellation was able to remove gold and silver from mixtures containing lead and other metals, but silver could not be removed. Gold parting as a process was specifically invented to remove the silver. The main ancient process of gold parting was by salt cementation and there is archaeological evidence of that process from the 6th century BC in Sardis, Lydia. In the post-medieval period parting using antimony, sulfates and mineral acids was also used. In the modern period chlorination using the Miller process, and electrolysis using the Wohlwill process are the most widely used methods of refining gold by removing silver and platinum. These latest processes have been employed for over 100 years. For some years now a new Acid Less Separation process, based on the vacuum distillation method, is being used; it is a physical method, which does not use any chemical and therefore it is considered the greenest of the gold parting processes.

Parting, in metallurgy, the separation of gold and silver by chemical or electrochemical means. Gold and silver are often extracted together from the same ores or recovered as byproducts from the extraction of other metals. A solid mixture of the two, known as bullion, or doré, can be parted by boiling in nitric acid. The silver is dissolved as silver nitrate, leaving a residue of gold that is filtered off and washed; silver is precipitated out of solution by the addition of ferrous sulfate. This is the traditional method used in assaying the content of gold and silver samples.

Most gold and silver are parted electrolytically after being recovered in the slimes left over from copper refining or as a metallic by-product of lead or zinc smelting. The bullion is cast into anodes, which are placed into an electrolytic cell and subjected to an electric current. Silver dissolves in the electrolyte and then deposits onto the cathodes. Gold and trace amounts of silver are recovered in the slimes and are parted either electrolytically or by boiling in sulfuric acid and potassium nitrate to dissolve the silver.

Kroll process

The **Kroll process** is a pyrometallurgical industrial process used to produce metallic titanium. It was invented in 1940 by William J. Kroll in Luxembourg.

Process

Former to the Kroll process, titanium was separated from its ores by the chloride process, where a feedstock of refined rutile or ilmenite is reduced with petroleum-derived coke in a fluidized bed reactor at 1000 °C. The product is then treated with chlorine gas, producing TiCl₄ and other volatile chlorides, which are then separated by continuous fractional distillation:

$$\begin{split} &\operatorname{FeTiO_3} + \operatorname{C} \longrightarrow \operatorname{Fe} + \operatorname{TiO_2} + \operatorname{CO} \\ &\operatorname{TiO_2} + 2\operatorname{C} + 2\operatorname{Cl_2} \longrightarrow \operatorname{TiCl_4} + 2\operatorname{CO} \end{split}$$

In the Kroll process, the TiCl₄ is reduced by liquid magnesium at 800–850 °C in a stainless steel retort to ensure complete reduction:

$$\mathrm{TiCl_4} + 2\,\mathrm{Mg} \longrightarrow \mathrm{Ti} + 2\,\mathrm{MgCl_2}$$

Complications result from partial reduction of the TiCl₄, giving to the lower chlorides TiCl₂ and TiCl₃. The MgCl₂ can be further refined back to magnesium. The resulting porous metallic titanium sponge is purified by leaching or heated vacuum distillation. The sponge is jackhammered out, crushed, and pressed before it is melted in a consumable carbon electrode vacuum arc furnace. The melted ingot is allowed to solidify under vacuum. It is often remelted to remove inclusions and ensure uniformity. These melting steps add to the cost of the product. Titanium is about six times as expensive as stainless steel.