A New Method of Processing Ilmenite for Titanium Compounds

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Summary Titanium tetrachloride containing less than 0·1% iron can be obtained from ilmenite by roasting and reduction with carbon, followed by chlorination.

ILMENITE is a mixed oxide, consisting mainly of titanium dioxide and iron(II) and iron(III) oxides. Several methods for the exploitation of ilmenite on a commercial scale to prepare titanium compounds[1] have the disadvantage that compounds of titanium and iron are produced together.

We have developed, on a laboratory scale, a process whereby titanium is selectively converted into a volatile compound. Finely powdered ilmenite was roasted in air above 1000°, so that the iron(II) oxide in it was oxidized to iron(III) oxide, as indicated by the development of a pale brown colour.

The roasted ilmenite was mixed with twice its bulk of powdered coke or coal and a suitable quantity of a catalyst (*e.g.* iodine). The mixture was heated in a closed furnace, out of contact with air, above 700°. Over 7 hr., a dull black colour homogeneously developed and a lumpy mass was formed. The solid mass, when cooled, was easily pulverised.

When the powder was heated with an equal quantity of ammonium fluoride[2] in a platinum vessel (the temperature was raised slowly to 500°) pure titanium tetrafluoride sublimed out. It was free from iron contamination. Titanium tetrafluoride can be reduced by calcium to titanium.

We found that chlorination of the powder obtained on carbon reduction of roasted ilmenite afforded a useful method of preparing $TiCl_4$. The powder was mixed with a small quantity of sodium chloride and iodine and heated

in a stream of dry chlorine. Titanium tetrachloride distilled at above 200—250°. It showed less than 0·1% iron contamination.

We are investigating the composition and characteristics of the iron carbide that was left behind. This has commercial potential of its own.

Titanium metal can be obtained from titanium tetrachloride by the Kroll process[3] or by reduction with sodium.[4]

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