Production of Titanium from TiCl_4 in an Arc Furnace

by L. D. Jaffe and R. K. Pitler

IT would clearly be advantageous to produce molten titanium, suitable for alloying and casting, directly from the relatively inexpensive tetrachloride, without using a metallic reducing agent. Accordingly, a preliminary investigation has been made of the production of titanium by hydrogen reduction of the tetrachloride in an electric arc furnace.

For the reaction:

Lockhart and his colleagues¹ have calculated equilibrium constants at 800° to 1500°K, based on tables of Brewer.² Extrapolating these data on the assumption that the heat of reaction varies linearly with temperature, and using Brewer's value for the heat of fusion, there is obtained for the reaction:

an equilibrium constant at 2100°K (100° above the melting point)

$$K_p = (p_{\text{HCl}})^4 / p_{\text{TiCl}_4} (p_{\text{H}_2})^2 = 0.167 \text{ atm.}$$

If this is correct, starting with hydrogen saturated with TiCl_4 at 25°C, where the vapor pressure of the latter³ is 12.6 mm, and maintaining the overall pressure at 1 atm, 99¼ pct of the TiCl_4 would be reduced to Ti at equilibrium.

An arc furnace designed for the melting of titanium and lined with water-cooled copper was used for the preliminary experimental check. Its inside diameter was 2 7/16 in. and height 6 in. The water-cooled electrode was tipped with ¾ in. diam tungsten. In the one run carried out, the furnace was charged with a starting batch of 293.35 g of scrap titanium,⁴ analyzing 99.24 pct Ti. After the system was twice pumped out and flushed with argon to remove air, the arc was struck in argon and kept at 400 amp dc, electrode negative, for 1 min to melt the starting batch. Commercial tank hydrogen, dried by passing over silica gel and bubbled through technical grade TiCl_4 at room temperature, was then admitted to the furnace. The inlet tube directed this gas downward toward the arc and melt; the gas outlet was at the top of the furnace. The quantity of gas passed through was not measured accurately but is estimated to be about 50 liters per min at a few mm of mercury above atmospheric pressure.

The arc was held in the H_2 - TiCl_4 mixture at 400 amp with little difficulty. After 10 min the arc burned through the copper lining above the melt, admitting water and ending the run. (This had happened frequently in straight melting runs under argon, and is not thought due to the atmosphere

used.) In the furnace were found an ingot, several small pieces, and a little metallic powder, evidently formed when water entered the hot furnace. Some titanium also clung to the electrode. The portions were weighed and separately analyzed for titanium. Their total weight was 321.76 g of 94.82 pct overall purity. Much of the impurity consisted of tungsten broken off when the titanium was removed from the electrode. No detailed impurity analysis was made because the starting materials were impure and flooding the furnace introduced further contamination.

Thus, the metal recovered contained 305.08 g Ti, as compared to 291.12 g in the starting batch. Titanium could not have been carried over from previous melting runs in the furnace, as the interior was sandblasted and the electrode ground clean before the run. Evidently at least 13.96 g of Ti were produced from TiCl_4 . Since this would require complete reduction of 430 liters of TiCl_4 gas at 12.6 mm of Hg, the efficiency of the process must have been rather high.

Experimental work is continuing. No reason is seen why the process could not be scaled up to the size of a 30-ton steel-melting arc furnace. In a large unit there would be a large molten pool from which liquid metal could be withdrawn for alloying and casting. The problems of electrodes and power supply are similar to those arising in arc furnaces for melting titanium. Unreacted TiCl_4 and the HCl in the exhaust gases could be recovered; unreacted H_2 could be dried and re-used.

The melt would be saturated with hydrogen, whose solubility in liquid titanium is unknown. Small amounts of hydrogen seem to have little effect on the properties of titanium.⁵ If the amount introduced was so large as to impair properties or cause casting difficulties, it could be reduced by holding the melt under an inert gas or by pumping on it, prior to casting.

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