

THE PRODUCTION OF FERROTITANIUM IN A
d.c. TRANSFERRED-ARC PLASMA FURNACE

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INTRODUCTION

Ferrotitanium is used as a carbide stabilizer in some special steels and, in particular, in ferritic stainless steel. Two ranges of grades of ferrotitanium are available. The lower grades contain between 20 and 50 per cent titanium and are produced by the aluminothermic reduction of ilmenite.¹ The higher grades contain between 65 and 75 per cent titanium and are produced simply by the co-melting of iron and titanium scrap. In the lower grades (which have a silicon content between 4 and 8 per cent), the ratio of titanium to aluminium is between 2 and 8.3; in the higher grades, it is between 15 and 150. The silicon contents of the higher-grade alloys range from 0.1 to 0.5 per cent.²

The grades with a nominal titanium content of 70 per cent are preferred, not only because of their higher titanium contents but because of their low melting points (1130 to 1200°C), which ensures their rapid assimilation when they are added to the steel during ladle processing.

Recent advances in methods for the remelting of titanium scrap to yield an acceptable grade of metal for aerospace application has resulted in a decline in the availability of this scrap for ferrotitanium production. This trend has renewed the search for alternative methods by which ferrotitanium can be produced

direct from mineral sources.

Some work had been done at the Council for Mineral Technology (Mintek) on the production of reactive metals by the aluminothermic reduction of their pure oxides.³ In the present study, however, the feasibility of the direct production of a high-grade ferrotitanium from a high-titania slag was investigated as part of Mintek's d.c. transferred-arc research programme. Mintek recognized the potential benefits of plasma technology some years ago, and currently has test facilities rated between 50 kVA and 3.2 MVA, which are used in investigations on the suitability of thermal plasma for a wide range of pyrometallurgical processes.

TRANSFERRED-ARC PLASMA FURNACES

The furnaces used during the present study consist essentially of a refractory-lined steel vessel fitted with an anode connection that penetrates the hearth refractories, a centrally positioned hollow graphite electrode that serves as the cathode and through which argon is passed to stabilize the arc, a roof with a stuffing box that closes the furnace and provides a seal round the electrode, and a readily controlled d.c. power source.

THE REDUCTION OF TiO₂

Compared with other, more expensive reactive metals, aluminium is the most practical agent for the reduction of titanium dioxide to titanium metal. The use of carbon results in the formation of carbide, and the Gibbs free energy, ΔG° , for carbide formation is more favourable, at all practical temperatures, than that for the formation of titanium metal. Reduction by silicon is not possible since the ΔG° for the reaction is positive at all practical temperatures.

The ΔG° for the reduction of titanium dioxide to titanium metal by aluminium is negative but, unfortunately, the ΔG° for the formation of

titanium monoxide is even more negative at similar temperatures. The free-energy change for the further reduction of this oxide is positive at the reaction temperatures and therefore the yield of titanium metal will be diminished to some degree.

Enthalpy Considerations

A number of metals are produced by aluminothermic reductions. The term 'aluminothermic' is usually taken to mean self-sustaining aluminium reduction reactions. In these processes, the enthalpy of reaction is high enough, i.e. at a level such that, after the reaction has been initiated in one part of the charge, sufficient thermal energy is liberated to heat progressively the rest of the reacting materials to a suitable reaction temperature and to superheat the metal and resulting slag to a temperature such that good separation of these two phases is achieved without the need of any other heat source.

Dautzenberg states that, for a self-sustaining reaction,

$\Delta H_{298}^{\circ} \leq -301$ kJ per gram-atom of aluminium.⁴ The reaction



has $\Delta H_{298}^{\circ} = -128.3$ kJ per gram-atom of aluminium and the ΔH_{293}° for the reduction of Fe_3O_4 and FeO is -418.2 and -440.3 kJ per gram-atom of aluminium respectively. This limits the titanium content of a ferrotitanium alloy that can be produced in a self-sustaining reaction to about 28 per cent.

Natural ilmenite of good grade, which contains some 46 per cent titanium dioxide, cannot be used for the usual practice involving self-sustaining reactions. However, if the ilmenite is pre-heated and fed to molten aluminium in a pre-heated reaction vessel, a self-sustaining reaction can be maintained.

Another option also exists for the production of higher-grade ferrotitanium alloys.⁵ Use can be made of chemical boosters such as barium peroxide or potassium perchlorate to generate the required extra thermal energy. This practice has the disadvantage that relatively expensive aluminium units are used essentially as a fuel to provide the energy requirement of the process.

Constitution of the Charge

The reaction of metal oxides by aluminium produces high-melting-point aluminium oxide. For good slag-metal separation, a flux must be added to lower the melting point of the remaining oxide phase. Lime is very suitable for this purpose because it forms a low-melting-point (1455°C) eutectic with aluminium at a lime-to-alumina ratio of 55:45. However, the use of lime results in the production of large volumes of slag. The amount of lime needed as a flux depends, of course, on the efficiency of the aluminothermic reaction. The presence of unreacted metal oxide in the slag can have a marked influence on the amount of lime needed to lower the liquidus temperature of the slag to a suitable level, as was found in the manufacture of ferrotitanium.

Choice of Raw Material

A local source of titania-rich slag containing 85 to 87 per cent titanium dioxide was available and it was decided that an attempt should be made to produce ferrotitanium from that source and to supply the shortfall in energy required for the completion of the reaction and the separation of the metal and the slag by electrical means. It was hoped that this would allow the optimum usage of aluminium as a reducing agent.

Reasons for the Choice of a d.c. Transferred-arc Plasma Furnace

Initially, attempts were made to produce ferrotitanium in a 60kW single-phase submerged-arc furnace. The operation was very difficult because

of the very high conductivity of the titaniferous slag. The penetration of the electrode into the bath could not be maintained at a constant level, and violent fluctuations occurred in the current drawn from the transformer. Operation in a semi-submerged-arc mode was necessary, but this raised the temperature of the top of the bath to a high level, and the reflected heat damaged the furnace roof. Also, the material in the lower part of the furnace tended to freeze and, thus, limited the amount of reaction that could take place.

Various ways in which the constituents of the charge could be introduced to the furnace were tried, but the operation did not improve. Although on most occasions some slag could be tapped from the furnace, the bulk of the product of the smelt had to be removed by digging-out the furnace when it had cooled. It was frequently found that large masses of metal were held up in the slag, and that contact of the electrodes with this metal probably caused the wild fluctuations in the current being drawn.

Although an alloy contained 61.6 per cent titanium, 9.0 per cent iron, and 15.1 per cent aluminium was produced in one test, it was felt that an arc-smelting operation would not be commercially viable, since it would be too erratic to be easily controlled, and that some other heating system, in which there would be no contact between the electrode and the charge, should be used. The transferred-arc plasma furnace appeared to offer an attractive alternative and, as a preliminary study, four tests of short duration were carried out in a 100 kVA furnace using ilmenite and the titania-containing slag as a source of titanium.

The tests demonstrated that the use of a transferred-arc plasma furnace for this particular reduction reaction was feasible. The operation of this furnace, unlike that of the arc furnace, was extremely stable. An alloy containing 31.2 per cent titanium was produced from ilmenite, while one containing 63.5 per cent

titanium was obtained from the titaniferous slag. The titanium content of this metal was similar to that obtained in the arc furnace except that the aluminium content was 7.9 per cent as against 15.1 per cent.

However, these preliminary tests did highlight some problems. It was not possible to tap metal from the furnace, and an examination of the contents of the furnace after they had cooled showed a poor slag-metal separation. The slag produced was seen to be very corrosive towards the magnesite lining of the furnace, and although, theoretically, the slag should have contained no magnesium oxide, it was found to contain 14 per cent.

SMALL-SCALE BATCH TESTS IN A 50 KVA WATER-COOLED FURNACE

The preliminary large-scale tests were done before the small-scale tests were started. However, the investigations were done in parallel at a later stage, and modifications to equipment and lessons learned in the small-scale tests were applied, wherever possible, in the large-scale tests.

Equipment and Procedures

The Mintek 50 kVA facility was used in these tests. A schematic diagram of the later development of this furnace is shown in Figure 1. The major features of this furnace are: the use of a pre-fired high-magnesia refractory crucible (200mm in diameter and 400mm in height) inside the water-cooled steel shell, an anodic connection consisting of a single steel rod (50mm in diameter) that penetrates centrally through the bottom of the crucible, and a pencilled electrode (25mm in diameter) that was to centralize the arc. The flowrate of argon through the electrode was 6 l/min.

In preliminary tests, there was considerable dissolution of the magnesia crucible into the slag. The reduction of this magnesia by the alu-

minium in the furnace charge resulted in low efficiencies of reducing agent and poor recoveries of titanium. Crucibles made from a range of other refractory materials (e.g. alumina, magnesia-titania, etc.) did not succeed in withstanding slag attack. This problem was overcome eventually by the use of the water-cooled jacket shown in Figure 1. This water-cooled jacket permitted a protective skull of frozen slag to form on the walls of the crucible. This approach was found to be highly successful, and no further problems with refractories were encountered.

The heat losses to the water-cooled jacket were monitored continuously as a precaution against any impending failure of the crucible refractory. These heat-loss measurements were also used as a means of operational control of the power requirements of the furnace. A typical test took approximately 1 hour, and the average arc power was about 30kW.

Objectives of the Experimental Work

The effects of the following variables were investigated during the small-scale tests:

- (1) the amount of lime added as a flux,
- (2) the aluminium added to the charge as a proportion of the stoichiometric requirement, and
- (3) the influence of the addition of iron to the charge.

Three separate series of tests were conducted, each series being dedicated to the study of one of the parameters mentioned above. The reproducibility of the results was found to be excellent in all cases, and titanium recoveries of over 50 per cent were obtained. Aluminium efficiencies (based only on the quantity of titanium reduced to metal) of up to 69 per cent were realized.

Effect of lime flux - As was mentioned earlier, lime is commonly used as a flux for the alumina generated during aluminothermic reductions. The formation of a slag with a low melting point

is particularly important in an autothermic process where the enthalpy of the reduction reactions has to provide the necessary thermal energy, not only to melt the slag, but to heat it to such a temperature that it has sufficient fluidity to ensure good disengagement of the metal and the slag phases. In an electric furnace, however, more refractory slags can be tolerated since the energy input is not dependent on process chemistry. Extended periods of superheating can also be used to improve slag-metal separation. A major incentive to the inclusion of smaller quantities of lime in the charge is the substantial reduction in slag volume that results. The lime addition required to give a calcium oxide-to-alumina ratio of 1 results in a slag-to-metal mass ratio of over 5 when the titania-bearing raw material that was selected is smelted. An appreciable quantity of thermal energy is therefore required to heat a material that is required only as a flux and is essentially inert in the reduction reactions. Some undesirable contaminants (e.g. carbon and silicon) that tend to concentrate in the ferrotitanium alloy are also introduced via the lime.

The effects of varying additions of lime flux to the smelting charge can be seen in Table I. An aluminium addition of 100 per cent of the stoichiometric requirement was used in these tests. This requirement was calculated on the basis of the total reduction of ferrous oxide, titanium dioxide, and silica contained in the titaniferous slag to their respective metals. The theoretical slag-to-metal ratio obtained with decreasing lime additions is also shown in Table I, and a significant decrease can be seen. Although the titanium content of the alloys produced remained relatively constant at about 55 per cent, it may be noted that a decrease in the quantity of lime in the charge results in a significant decrease in the residual aluminium level in the alloy. The test in which no lime was used (run no. 4) resulted in the highest titanium-to-aluminium mass ratio being obtained in the ferrotitanium alloy (i.e. 3.1).

Effect of substoichiometric aluminium addition - In the second series of tests, the influence of smelting under substoichiometric reducing-agent conditions was examined. No lime was used in these tests. The results obtained for four different substoichiometric levels of aluminium are given in Table II, from which can be seen that, although the ratios of titanium to aluminium in the ferrotitanium alloy may be improved by smelting under substoichiometric reducing-agent conditions, this improvement is accompanied by a rise in the oxygen content of the alloy. Mineralogical examination of the slags produced in these tests revealed the presence of suboxides of titanium (TiO , Ti_2O_3), which had resulted from the partial reduction of titanium dioxide.

Effect of iron addition - A series of experiments was carried out in which metallic iron was included in the smelting charges. No lime was used in these tests. These tests were done in an attempt to determine the influence of added iron on the ratio of titanium to aluminium in the alloys produced. The results of this series of tests are shown in Table III. Metallic iron in the form of metallic flakes constituted 10 per cent (by mass) of each charge. An immediate benefit realized from this practice was a significant improvement in the slag-metal separation. This is attributed to the increase in density of the ferrotitanium, which results from increased iron concentrations in the alloy. The average relative density of alloys produced in the first two series of tests was only 4.4 compared to an average value of 5.4 obtained in the third series. Table III shows that the inclusion of iron in the smelting charge has very little effect on the ratio of titanium to aluminium.

Interpretation of Results of the Small-scale Tests

The substantial decrease in the slag-to-metal ratio that occurred when no lime was added to the smelting charge resulted in more efficient heat transfer to the lower regions of the bath.

In the particular furnace configuration used in these tests, most of the arc energy is dissipated on the surface of the bath at the point of arc attachment.⁶ As titania slags have high electrical conductivity, it may be assumed that the degree of resistance heating in the slag is negligible. This improved energy transfer ensures that the metal in the lower regions of the bath remains fluid and is therefore able to participate in the reactions throughout a run. This effect, coupled with the higher reaction temperatures that prevail during operation with more refractory slags, is believed to be one of the reasons for the improvement in the ratio of titanium to aluminium in the alloy as the lime additions to the charges were decreased. Another factor may be the increased activity of the titanium dioxide in the slag, which is suppressed at high concentrations of lime.

Smelting with substoichiometric quantities of aluminium leads to a decrease in the residual aluminium in the alloys produced due to the more favourable reaction equilibria that are established under these conditions. However, it appears that a thermodynamic barrier is encountered that does not allow ferrotitanium alloys with a titanium-to-aluminium mass ratio higher than about 7 to be produced. It appears that attempts to improve this ratio by further decrease of the aluminium contained in the charge result only in a corresponding increase in the oxygen content of the alloy.

LARGE-SCALE CONTINUOUS TESTS

The transferred-arc plasma furnace used in the large-scale tests is shown in Figure 2. A graphite electrode of 50mm diameter with a 10mm axial hole for the argon is used in the furnace. The design of the roof seal is similar to that of the 50 kVA furnace. The anodic connection is via three 50mm steel rods, which enter the bottom of the furnace and initially protruded some 20mm through the hearth refractory. As the project progressed, the furnace was modified. The major changes were as follows. The moulded, domed,

alumina refractory roof was replaced by a flat steel roof, which was faced with a layer of magnesite refractory and, on its hot face, with a layer of baked electrode paste. Finally, an alumina-brick lining was installed, and provision was made for limited water cooling on the furnace shell.

The charge was fed to the furnace via a totally enclosed vibratory feeder which was so designed as to minimize the ingress of air. The furnace was initially powered by a 100kW power source, which consisted of a three-phase transformer and a water-cooled diode rectifier. At a later stage, a thyristor-controlled system, which is nominally rated at 200kW and is part of a larger plasma-furnace installation, became available and was used. This larger facility was used purely for convenience; operation of the unit was easier, and a higher power level could be achieved.

The primary objectives of the first campaign were: the establishment of an acceptable mass balance, improvement of the grade of alloy produced in terms of its titanium and aluminium contents, and determination of the efficiency of utilization of the aluminium in larger-scale operation.

Table IV gives the composition of the metal recovered from the hearth after an extended test in which six consecutive batches of feed were charged to the furnace but only a few hundred grams of metal were present in the slag for each tap.

Table V shows the recovery and accountabilities of the titanium, iron, and aluminium.

The efficiency of aluminium usage in the reduction of titanium dioxide and silica by aluminium was calculated as being only 45.5 per cent. Severe erosion of the magnesite refractory was a feature of this campaign, and an unexpected excess of alumina was found in the slag. This, it was presumed, was due to the reduction of mag-

nesium oxide that had been dissolved from the furnace lining. The magnesium that formed was volatilized and then re-oxidized at the mouth of the gas offtake, giving rise to copious white fumes. Confirmation that this is what had occurred was obtained when a sample of fume was collected at a later stage. Analysis showed that the major constituent of the fume was magnesium oxide (64.4 per cent).

An attempt was made to improve the quality of the magnesite lining by sintering of the lining with the arc before use. The arc was struck on a graphite block placed on top of the anodes in the furnace. No improvement was noted, and the slag produced still contained 14 per cent magnesium oxide.

For this particular test, the aluminium in the charge had been reduced to 90 per cent of the stoichiometric requirement. The metal produced had a titanium content very similar to that produced in the small-scale test under similar conditions, but the titanium-to-aluminium ratio was 8.1, which is a great improvement.

At that stage, it was decided that the furnace should be relined with alumina bricks. It was hoped that, because of their more dense structure, these bricks would better withstand the corrosive action of the slag. A test of short duration was conducted so that the performance of the alumina refractory could be observed. It was soon obvious that even this material was inadequate because severe undercutting was seen at the slag line.

Because the small-scale tests had shown that water cooling of the refractory was an effective means whereby slag attack could be avoided, a water-cooled circumferential panel was fitted to the furnace shell. The area that could be cooled was limited in extent because of the design of the furnace shell, but was adequate since it covered the most critical area.

FURTHER EXPERIMENTAL WORK

A further campaign was then mounted so that this modification could be tested, and some of the operating conditions were changed. Because the higher-powered thyristor drive was being used at that stage, a higher feed rate and an increased pro-rated power were used in an attempt to shorten the time of reaction and, hence, to minimize the time for reaction between the alloy formed and the air. The lime addition was decreased by half, and the aluminium addition was reduced to 80 per cent of the stoichiometric requirement.

Control of the smelting operation was easy at this higher power level but, although the slag was readily tapped, the metal still remained in the furnace. This accumulation of metal (and entrained slag) gradually caused the level of the bath to rise, which increased the thermal load on the furnace roof. Attempts to flush out the accumulation of the addition of extra lime and titania slag met with only moderate success.

The composition of the first 10 slags tapped from the furnace is given in Table VI.

The composition of the samples of metal dug from the furnace hearth is given in Table VII.

It is obvious from the wide fluctuations in composition of the slags that this campaign suffered from a lack of metallurgical control despite careful monitoring of the feed rate and rate of energy input. The titanium content of the metal dug out of the hearth was low, except for sample 1, but the titanium-to-aluminium ratio was encouragingly high in some cases. The oxygen content of the metal was also high, but it was felt that this could have been partly due to slag entrainment as well as to the effect of a reduced aluminium addition.

MELTING POINT OF THE ALLOY

At that time, some attention was given to

the liquidus temperature of the alloy being produced. Samples of alloy were selected, and attempts were made to melt them in a graphite resistance furnace under an argon atmosphere. The samples were contained in alumina crucibles, and the furnace was so constructed that the samples could be observed while being heated. The temperature was measured by means of an optical pyrometer. The temperature at which the sample slumped was taken as the melting temperature.

The melting temperatures together with the composition of the samples, including their oxygen and nitrogen contents, are shown in Table VIII. It can be seen that, except for sample 5, which had a high aluminium content, the melting points were in excess of 1700°C. This, no doubt, was the reason why the metal froze in the furnace. The high liquidus temperature is probably attributable to the oxygen and nitrogen contents of the alloy.

THE ADDITION OF IRON TO THE CHARGE

The application of water cooling to the furnace shell successfully stopped slag attack on the refractory. However, it had still not been possible to tap an appreciable quantity of alloy from the furnace.

A further campaign was conducted, therefore, in which a quantity of iron was included in the charge. The amount of iron added was equal to 40 per cent (by mass) of the titania-rich slag in the charge. The objective was to lower the liquidus temperature of the alloy by diluting it with iron, and to improve the slag-metal separation by increasing the density of the alloy.

This was only moderately successful and, while slag and some metal was tapped from each heat, there was again a build-up of material in the furnace. The composition of the metal tapped from heats 5 to 15 is shown in Table IX, which indicates that a fairly consistent grade of metal was produced. The oxygen content and the nitrogen content were also considerably reduced.

The melting temperature of the metal produced was determined to be about 1500°C.

It was calculated that the recovery of titanium as metal in the tapping tray was only 40 per cent. The rest remained in the furnace, entrained in the slag.

After being allowed to cool, the furnace was dug out, and the major proportion of the frozen material was recovered as a solid ball. This ball had an outer layer of black slag, which had formed against the furnace walls, and a mass of greenish-yellow material and some layers of silvery metal (which were later shown to be iron) in the centre.

Mineralogical examination of the greenish-yellow slag showed that the material contained metal globules in a slag matrix. Each of these globules contained three phases, each with a different titanium-to-iron ratio. There was also a fine dispersion of yellow material throughout the slag. This was determined to be titanium nitride.

It was obvious from this that the exclusion of air from the furnace was inadequate, even though attempts had been made to control it.

FURTHER FURNACE MODIFICATIONS

A further campaign was conducted in which a far greater effort was made to control the ingress of air to the furnace. The gas offtake was modified, and the joint between the roof and the body of the furnace was caulked with refractory cement. Attention was also given to the electrode seal in the stuffing box. In addition, the furnace was fed at a high rate so that the time available for reaction of the metal with nitrogen or oxygen would be as short as possible.

These attempts were in vain, since it was still not possible to tap metal from the furnace. However, the dig-out revealed no signs of titanium

nitride in the slag. Nevertheless, it was clear that major redesign of the furnace and feeding system was necessary if success was to be achieved.

SMALL-SCALE SEALED FURNACE

A small-scale (50kW) sealed plasma furnace, which had been developed from the pot-test facility, was available, and it was decided that tests should be conducted in that furnace before the 200kW facility was modified.

Figure 3 shows the design of the furnace, which uses the same type of magnesia crucible as that used in the other small-scale experiments. The aim in these tests was: determination of the oxygen and nitrogen contents of the alloy produced under air-free conditions as well as the effect of iron additions on the concentrations of these elements. Table X compares the composition of the metal produced in three tests in the sealed furnace with that of three alloys produced in the unsealed furnaces but having similar titanium and iron contents.

It can be seen that smelting in a sealed furnace has a significant influence on the nitrogen content of the alloy produced, but does not appear to influence its oxygen content. The oxygen content appears to be a function of the equilibria reached by the reacting species.

CONCLUSIONS

Alloys of titanium and iron have been produced by the reduction of a titania-rich slag with aluminium in a d.c. transferred-arc plasma furnace of the molten-anode configuration. Tests were conducted at 50 kVA and 200 kW. A typical ferro-titanium alloy that can be produced contains 56.5 per cent titanium and has a titanium-to-aluminium ratio of 4.2.

Factors such as the amount of lime flux added as well as the proportion of the stoichiometric addition of aluminium have an effect on the ratio of titanium to aluminium and, in both cases, a

decreased addition improves this ratio. However, at a lower aluminium addition, the recovery of titanium is lower and the residual oxygen content of the metal is higher.

The residual oxygen and the nitrogen have a very marked and deleterious effect on the liquidus temperatures of the metal. It had been thought that, because of the design of the furnace, the use of argon as a plasma gas, and the large slag volumes, the alloy would be protected from the nitrogen in the atmosphere. However, the results show that this was not so, and that an especially sealed furnace was needed to produce an alloy containing less nitrogen. The use of this furnace resulted in a tenfold decrease in the nitrogen content of the alloys, but the oxygen content still ranged between 3 and 5 per cent. This shows that the oxygen content is not primarily a function of oxidation by the atmosphere but of the degree to which the reduction reaction proceeds, i.e. at whatever concentration of reactants and products equilibrium is reached.

This study has shown that the aluminothermic reduction of titania-rich slag to meet a titanium-to-aluminium specification is probably not practically or theoretically feasible in a single-stage operation. Two options appear to be possible, as follows.

(1) Excess aluminium could be used to form a titanium-aluminium alloy with an oxygen content sufficiently low for the alloy to be tapped. (This practice should enhance the recovery of the titanium.) The aluminium would have to be removed subsequently, by, for example evaporation in an electron-beam or plasma furnace.⁷ This approach would probably be very costly, and would not be economically viable while adequate supplies of titanium scrap are available.

(2) The influence of iron additions on the liquidus temperatures of alloys (containing 30 to 40 per cent titanium and having an improved titanium-to-aluminium ratio and, it is hoped,

a lower oxygen content) needs to be investigated on the larger scale in a sealed furnace. This approach seems to be the one most likely to succeed.

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Fig. 1 - Schematic representation of the 50 kVA water-cooled batch furnace. (1) Argon supply to hollow graphite electrode, (2) Electrode support mechanism, clamp, and power cable, (3) Water-cooled electrode seal, (4) Gas offtake, (5) Steel roof with magnesia lining, (6) Ceramic fibre insulating gaskets, (7) Water-jacket, (8) Replaceable refractory crucible, (9) Anode connection, (10) Graphite block, (11) Feed port.

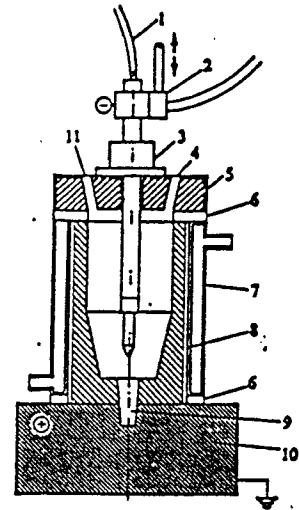


Fig. 2 - Schematic representation of large-scale furnace (nominally 200kW). (1) Argon supply to hollow graphite electrode, (2) Electrode support mechanism, clamp, and power cable, (3) Feed chute from sealed feeder, (4) Domed refractory roof (later replaced with a steel-backed, electrode-paste hot-faced magnesia roof), (5) Rammed magnesia lining (later replaced by alumina brick lining), (6) Water-cooled panel (fitted at later stage), (7) Anodes, (8) Tap hole launder, (9) Gas offtake, (10) Water-cooled electrode seal. Dimensions of crucible: Depth 600mm (later increased to 750mm), Diameter 500mm.

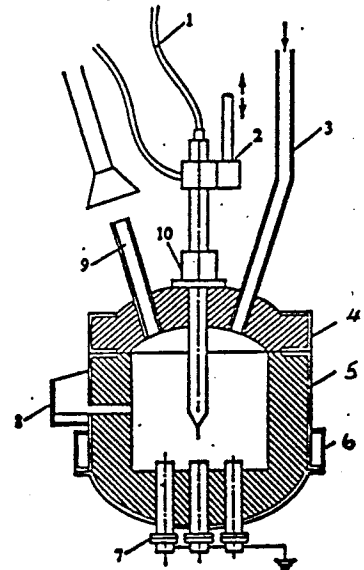


Fig. 3 - Schematic representation of sealed furnace. (1) Argon supplied to water-cooled hollow copper-graphite composite electrode, (2) Electrode support mechanism, clamp, and power cable, (3) Stainless-steel bellows and electrode seal, (4) Observation window, (5) Off-gas system with water-cooled baffle box, (6) Off-gases to water seal, (7) Replaceable magnesite crucible with anodic connection through bottom, (8) Graphite plate with external anodic connection, (9) Refractory-lined bottom portion of furnace, (10) Water-cooled middle section of furnace, (11) Refractory lined upper portion of furnace, (12) Feed pipe from sealed hopper.

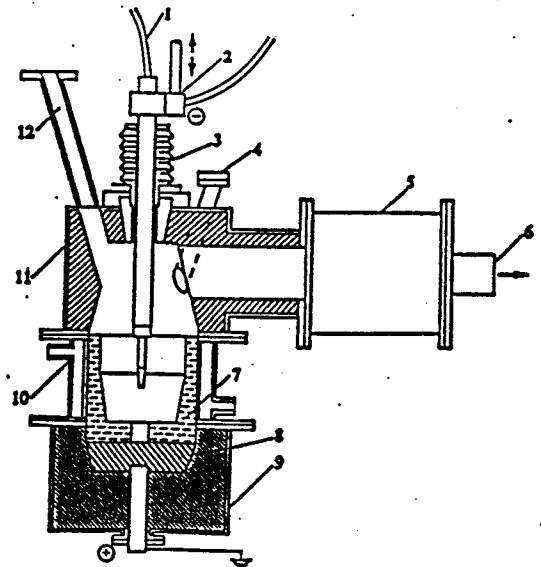


Table I - Small-scale Batch Tests: Effect of Lime Flux

Run	Lime addition as CaO:Al ₂ O ₃ mass ratio aimed for	Slag/metal mass ratio	Chemical composition of metal produced (% by mass)								Chemical composition of slag produced (% by mass)		
			Ti	Fe	Al	Si	O	N	C	Ti/Al mass ratio	Ti	Al ₂ O ₃	CaO
1	50:50	5.2	55.4	9.42	25.6	3.03	1.3	0.95	0.57	2.2	13.6	37.2	36.7
2	40:60	3.6	54.4	13.7	21.7	2.85	1.8	0.30	0.19	2.5	13.7	45.0	32.5
3	30:70	2.4	57.2	10.0	21.3	1.63	2.7	0.26	0.06	2.7	14.5	54.5	27.3
4	0:100	1.5	54.8	11.5	17.9	1.89	2.5	0.42	0.09	3.1	18.5	73.5	< 0.1

Table II - Small-scale Batch Tests: Effect of Substoichiometric Aluminium

Run	Aluminium as percentage of stoichiometric requirement	Chemical composition of metal produced (% by mass)						Chemical composition of slag produced (% by mass)	
		Ti	Fe	Al	O	N	Ti/Al mass ratio	Ti	Al ₂ O ₃
1	90	57.6	10.6	14.0	2.9	0.52	4.1	18.7	78.4
2	80	58.8	11.9	10.4	3.6	0.29	5.7	22.3	70.0
3	70	57.2	11.9	9.78	2.8	0.42	5.9	24.9	64.8
4	60	51.4	14.0	7.9	12.6	0.52	6.5	30.6	62.0

Table III - Small-scale Batch Tests: Effect of Iron Additions

Run	Aluminium as percentage of stoichiometric requirement	Chemical composition of metal produced (% by mass)				Chemical composition of slag produced (% by mass)	
		Ti	Fe	Al	Ti/Al mass ratio	Ti	Al ₂ O ₃
1	90	44.0	35.1	13.9	3.2	16.7	83.2
2	80	45.1	35.4	10.1	4.5	24.1	73.7
3	70	41.5	40.0	6.79	6.1	26.8	67.6
4	60	40.2	42.2	6.54	6.1	32.1	63.3

Table IV - Large-scale Test No. 1: Composition of the Metal

Ti %	Fe %	Si %	Al %	Ti:Al Mass ratio
56.4	14.5	2.8	13.5	4.1

Table V - Large-scale Test No. 1: Recovery and Accountability of Titanium and Aluminium

Element	Metal %	Slag %	Accountability %
Ti	46.5	40.1	89.1
Fe	81.2	60.9	142.1
Al	11.1	64.6	79.8

Table VI - Large-scale Test no. 2: Composition of the Slags

Slag no.	Origin	TiO ₂ %	FeO %	SiO ₂ %	CaO %	MgO %	Al ₂ O ₃ %	Mass %
1	Heat 1	64.8	0.65	2.22	7.8	0.60	26.1	31.6
2	Heat 2	38.3	1.00	1.30	16.6	0.60	47.8	29.1
3	Heat 3	27.8	0.18	1.40	13.8	0.52	60.2	42.2
4	Heat 4	36.0	3.33	0.68	15.8	0.59	48.4	27.0
5	Heat 5	33.0	0.28	0.32	17.6	0.60	51.0	25.6
6	Heat 6	25.9	0.97	1.92	13.8	0.50	52.8	24.1
7	Heat 7	29.5	0.72	1.00	11.0	0.52	60.0	31.0
8	Heat 8	47.0	1.70	0.95	9.4	0.60	45.0	20.1
9	Heat 9	30.2	1.12	1.60	15.3	0.42	53.1	39.8
10	Heat 10	33.0	0.55	0.55	22.6	0.68	47.0	24.8

Table VII - Large-scale Test no. 2: Composition of Metal Samples from Hearth

Sample no.	Ti %	Fe %	Al %	Si %	O ₂ %	Ti/Al Mass ratio
1	61.7	7.8	5.8	0.8	8.0	10.6
2	43.9	30.5	5.5	3.9	6.0	8.0
3	52.8	21.5	3.5	3.3	9.5	15.1
4	53.9	21.5	3.1	3.1	7.7	17.4
5	50.4	18.8	5.5	2.8	10.6	9.2

Table VIII - Composition and Liquidus Temperatures of Selected Alloys

Sample no.	Ti %	Fe %	Al %	Si %	O ₂ %	N ₂ %	Liquidus temp. °C
1	48.3	16.1	13.5	2.86	13.5	-	> 1740
2	61.7	7.82	5.86	0.81	8.0	-	> 1720
3*	71.0	21.4	3.2	0.1	0.95	0.38	1200
4†	53.9	11.7	20.2	0.7	3.6	-	1406
5	55.4	9.42	25.6	3.03	1.3	0.95	1507
6	60.0	9.65	12.9	1.18	5.2	0.84	> 1700
7	57.6	10.6	14.0	0.95	2.9	0.52	> 1700
8	27.5	64.8	5.0	0.9	0.6	0.03	1503
9	31.4	56.5	6.3	1.7	0.3	0.03	1489
10	59.0	14.8	10.8	3.15	6.0	2.8	> 1700
11	56.6	17.3	6.1	2.2	3.7	0.3	> 1700

*Commercial 70 per cent alloy

†Alloy made in argon atmosphere

Table IX - Large-scale Test no. 3: Composition of Tapped Alloy

Heat no.	Ti %	Fe %	Al %	Si %	O ₂ %	N ₂ %	Ti/Al Mass ratio
5	24.4	69.1	2.41	1.32	0.42	0.02	10.1
6	24.9	68.5	3.50	1.16	0.29		7.1
7	24.7	67.0	3.78	1.13	0.48		6.5
8	24.8	67.1	3.74	1.24	0.63		6.6
9	24.8	64.9	4.23	1.15	0.39		5.8
10	24.2	68.5	3.72	1.18	0.56	0.02	6.5
11	25.5	67.6	3.43	1.10	0.44		7.4
12	24.0	69.4	3.07	1.20	0.64		7.8
13	25.7	66.3	3.49	1.04	0.30		7.4
14	13.2	82.6	0.88	0.72	0.82		15.0
15	27.0	64.6	4.84	1.01	0.79	0.05	5.6

Table X - Comparison of the Composition of Metals Produced
in the Sealed and Open Furnaces

Furnace	Feed recipe	Ti	Al	Fe	Si	O	N	Liquidus temp. °C
		%	%	%	%	%	%	
A	100% Al	56.8	14.7	14.0	2.2	4.8	0.04	< 1580
A ¹		55.0	17.0	11.0	1.8	8.0	1.3	
B	100% Al+8.5% Fe	49.0	12.2	28.7	2.0	3.2	0.03	< 1650
B ¹		48.0	8.9	27.7	1.6	4.0	0.51	
C	100% Al+10% Fe	46.7	8.1	34.0	2.7	3.0	0.04	< 1650
C ¹		45.1	10.1	35.4	1.0	2.3	0.12	

A, B, C Sealed furnace.

A¹, B¹, C¹ Open furnaces