# Production of Al-Ti master alloys from TiO$_2$ in a DC arc furnace

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### Synopsis

The production of a 40% Al-Ti alloy was successfully proven at the lab scale, using pure TiO$_2$, aluminium, and lime. These tests were conducted in an induction furnace maintained at 1600–1625OC. The introduction of rutile into the feed recipe resulted in an alloy containing between 0.35 and 1.10% Zr, depending on rutile to TiO$_2$ ratio.

DC arc production of the alloy was largely accomplished at 40 and then 200kW scale. These tests evaluated various metallurgical and operational factors including target slag composition, Ti-content in the alloy, co-feeding versus sequential, furnace bath depth, and rutile as part of the Ti containing feedstock. Overall, an alloy containing 24–33%Ti was consistently produced with co-feeding of the reactants and with lower taphole tapping. When rutile was added, the zirconium analysis in the alloy remained below 0.2%. The main impurity elements were iron, silicon, carbon, nitrogen, and oxygen.

### Introduction

Aluminium titanium master alloys are largely produced by reacting halide salts with molten aluminium[1]. The reaction is carried out in a gently stirred furnace (either electric resistance, but mostly in an induction heating furnace) that is maintained at temperatures between 750–1200°C, depending on the desired Ti content of the final alloy. The overall reaction can be represented as:

$$13A1 + 3K_2TiF_6 = 3A1_3Ti + 3KA1F_4 + K_3A1F_6 \qquad [1]$$
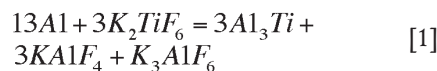
Excess aluminium is always present so that the final product contains 2–10%Ti. After decanting the produced salt, the alloy is vigorously stirred for a limited period of time in order to evenly disperse the Al$_3$Ti particles into the aluminium matrix. The alloy is then cast into various shapes and sold to aluminium producers.

Boron can also be introduced into the master alloy. This is usually accomplished by adding KBF$_4$:

$$2KBF_4 + 3A1 = A1B_2 + 2KA1F_4 \qquad [2]$$

In the presence of Al$_3$Ti, aluminium, di-boride is converted to titanium di-boride according to:

$$A1B_2 + A1_3Ti = TiB_2 + 4A1 \qquad [3]$$

The above reactions take place at the metal/salt interface, and rely on the diffusion of the reactants and products to/from the reaction site, which is induced by stirring. Diffusion is also enhanced by the exother-micity of the reactions as it causes local temperature gradients.

Another commercial process for Al-Ti production involves the addition of pure titanium into a molten bath of aluminium[2]. A protective salt layer is maintained in order to minimize the oxidation of the alloy. This process is not widely used, as compared to the halide salt route, apparently due to high metal losses and, thereby cost considerations.

In general, the commercial processes suffer from the following drawbacks:

➤ Environmental considerations including fluorine emissions and treatment and disposal of the resulting 'slag'
➤ The alloy tends to trap slag particles requiring vigorous stirring for their removal. Excessive stirring tends to increase the particle size of Al$_3$Ti particles (as well as TiB$_2$ when present), and thus reduces the effectiveness of the master alloy as a grain refiner, and results in metal losses due to oxidation
➤ Titanium recovery to the alloy is relatively low and ranges from 70–75% only
➤ Therefore, the production costs are very high, particularly when pure metals are used.
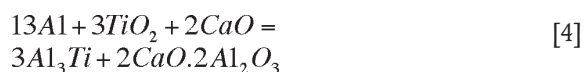
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In order to overcome some of these issues, researchers have suggested the use of pure titanium dioxide, high quality rutile[2], or even sponge titanium[3] as the source of titanium. In these processes, the preparation of the AlTi master alloy is carried out under a protective salt layer in order to minimize the oxidation of the alloy. This is more critical when titanium dioxide is employed as the Ti-containing feed material, as it requires an operating temperature of 1000–1200°C. It is reported that when titanium dioxide is employed, the reaction is relatively fast (being completed in10–15 minutes), but the limited solubility of TiO$_2$ in the salt (5–7%) is a concern. The 'slag' produced is a mixture of halides and oxides, where high Al$_2$O$_3$ content can decrease the solubility of TiO$_2$ even further (2). Although this approach has the potential of reducing the production costs, it does not completely address the environmental concerns. In addition, Ti-recovery may not reach 70–75%.
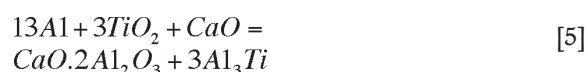
Mintek has been involved in the development of a process for the production of AlTi alloys since the early part of this decade. The important features of the process include:

➤ Aluminothermic reduction of titanium dioxide (pure TiO$_2$, rutile, etc) in a DC open arc furnace
➤ The process is carried out at relatively high temperatures (1600–1700°C)
➤ Dead burnt lime is employed to flux the Al$_2$O$_3$ produced by the reduction reactions. The slag, therefore, can be disposed of safely without the need for treatment
➤ The target titanium content in the alloy is 35–40%Ti. This alloy can be diluted to the desired composition by mixing it with molten aluminium, which is then used as a grain refiner in the aluminium industry. High Ti containing alloy has the potential of reducing the dosage levels and possibly the costs to the user
➤ The process has the potential of being carried out on a continuous basis.

Depending on the feed composition and the operating temperature, the overall chemical reaction can be expressed as:

$$13Al + 3TiO_2 + 2CaO = \qquad\qquad [4]$$
$$3Al_3Ti + 2CaO.2Al_2O_3$$

or;

$$13Al + 3TiO_2 + CaO = \qquad\qquad [5]$$
$$CaO.2Al_2O_3 + 3Al_3Ti$$

This paper presents the experimental work that has been undertaken so far, and briefly discusses the metallurgical results.

## Experimental equipment and procedures

Laboratory-scale tests were carried out in a medium frequency induction furnace. These tests aimed at evaluating the chemistry of the process and testing various feed recipes. The tests were carried out at 1600–1625°C using either lumpy or pulverized feed mixtures. The working crucibles were made of recrystallized alumina and varied in size

depending on the batch mass (100–300 g). After reaching the target temperature, the raw materials were allowed to react for an additional 30–60 minutes under argon atmosphere.

A 40 kW DC arc furnace was then employed in order to prove the process at a larger scale thereby generating a preliminary materials balance. The facility was operated by feeding a premix of the raw materials at 15 kg/h where the batch mass was kept at 15 kg. The operating power was 30–35 kW, resulting in a tapping temperature of roughly 1650°C. The products were tapped by either tilting the furnace, drilling through the taphole, or by oxygen lancing.

Finally, production trials were conducted in a 200 kW DC arc facility (Figure 1). The testwork was carried out at 100–160kW depending on the total feed rate (75–200 kg/h), feed mode (sequential or co-feeding), energy losses, etc. The target temperature was 1600–1650°C and the batch mass was 100 kg. Notice that both furnaces were lined with alumina castable.

Throughout the testwork, the metal and slag compositions were selected so that the densities are about 3.2–3.3 g/cm$^3$ and 2.8–3.0 g/cm$^3$, respectively, among other factors. The target slag composition was aimed at prompt tapping of the furnace products at the intended operating temperatures, although it was expected that refractory erosion would take place. The use of water cooled shell in certain tests was thought to be adequate in order to minimize refractory attack. The testwork was guided by thermo dynamic simulations using Pyrosim software package.

## Raw materials

Table I shows the chemical analyses of the raw materials used in the various stages of the testwork. Pigment grade titanium dioxide (TiO$_2$) was acquired from Servochem, a
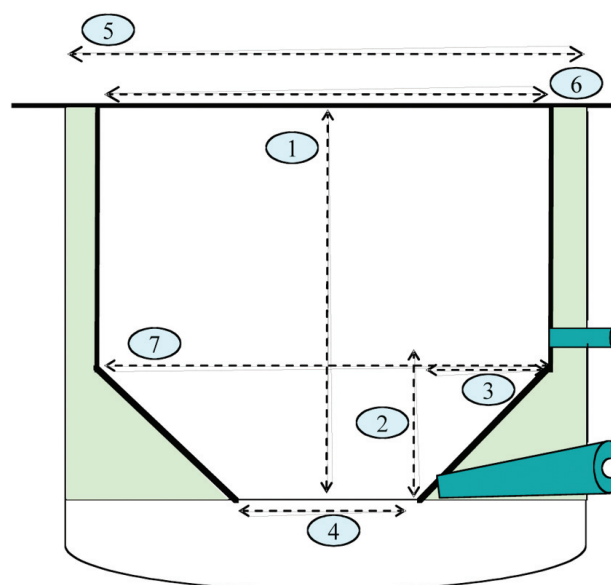


**Figure 1—Schematic of the 200 kW DC arc furnace. 1: 600 mm, 2: 170 mm, 3: 150 mm, 4: 455 mm, 5: 970 mm, 6: 645 mm, 7: 620 mm**

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### Table I

**Chemical analyses of the raw materials, mass per cent**

| Component | TiO$_2$ Pigment | Rutile | Lime | Aluminium | |
|---|---|---|---|---|---|
| | | | | Element | Mass % |
| MgO | NA | <0.01 | 1.745 | Mg | 0.01 |
| Al$_2$O$_3$ | NA | 0.50 | 0.735 | Al | 99.67 |
| SiO$_2$ | <0.1 | 2.15 | 2.029 | Si | 0.09 |
| CaO | NA | 0.05 | 93.00 | Ti | 0.01 |
| TiO$_2$ | 99.5 | 94.5 | 0.069 | Fe | 0.15 |
| Fe$_2$O$_3$ | NA | 0.55 | 0.372 | Pb | 0.01 |
| V2O5 | NA | 0.35 | 0.05 | Cu | 0.01 |
| ZrO$_2$ | 187 ppm | 0.70 | 9ppm | Zn | 0.01 |

NA: not available

South African commodity trader (Grade A-HR). It has a particle size of 40 μm and a bulk density of 0.87 g/cm³. Rutile, with a particle size of 115 μm and a bulk density of 1.84 g/cm³, was obtained from a South African producer. Aluminium (5 μm powder, or 5-10 mm pellets) was employed as the reductant (Zimalco). Lumpy lime (-40 mm, Lime Distributors) was used to flux the produced alumina.

## Results and discussion

The Lab-scale tests were aimed at determining the effect of Al/TiO$_2$ mass ratio in the feed on titanium extraction and the alloy composition (Figure 2). Clearly, titanium recovery was systematically high at all levels of aluminium addition, reaching more than 99.8% when Al/TiO$_2$ ratio was 4.5/1. The titanium analysis in the alloy dropped gradually with increased aluminium in the feed. Figure 2 suggests that an Al/TiO$_2$ ratio of 1.8 and lower is required in order to produce an alloy with 35% Ti and higher.

Figure 3 presents the analyses of zirconium, silicon, and titanium in the resulting alloy as a function of rutile content in the Ti containing feed stock. The results pertaining to 0% rutile addition are the average values of four different tests carried out at the same conditions. Based on CES specifications (Table II), rutile addition of up to 40% might be suitable in order to limit zirconium and silicon levels to the specified maximum upon dilution of the alloy to the 10% Ti-grade.

It should be noted, that aluminium addition was adjusted in order to maximize, in part, the density difference between the metal and the slag for effective separation. However, one advantage of increased aluminium addition is to minimize the oxygen content in the alloy (Figure 4), provided that air ingress is largely prevented and that the reaction is allowed to approach completion. These results pertain to tests (1–2kg batches) carried out in a 40 kW DC arc furnace using water-cooled copper crucible. Aluminium addition was varied in
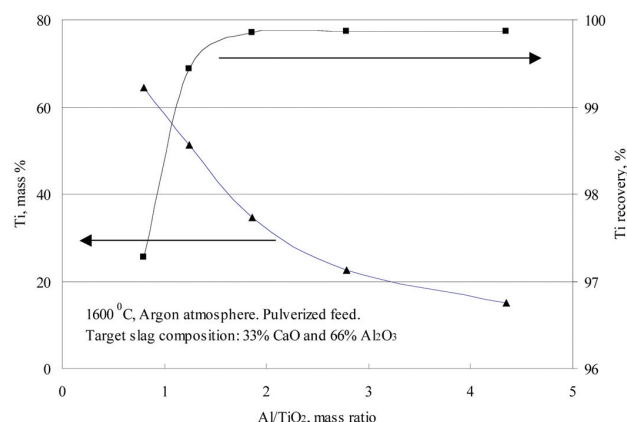


**Figure 2—Ti analysis in the alloy and its extraction as a function of Al/TiO$_2$ ratio in the feed**
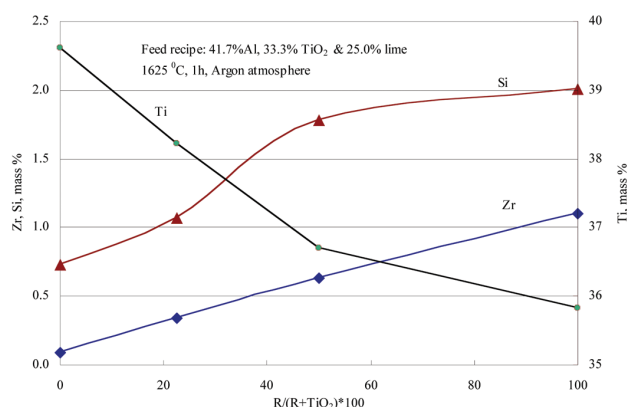


**Figure 3—Ti, Zr, and Si contents in the alloy versus rutile/(rutile+TiO$_2$) mass ratio in the feed**

### Table II

**CEN* chemical specification limits for impurities in 10% Ti-Al alloy, mass per cent[6]**

| Si | Fe | Cu | Mn | Mg | Cr | Ni | Zn | V | Others |
|---|---|---|---|---|---|---|---|---|---|
| 0.3–0.5 | 0.3–0.7 | 0.2 | 0.4 | 0.5 | 0.05–0.10 | 0.05–0.20 | 0.3 | 0.3–0.5 | 0.05 |

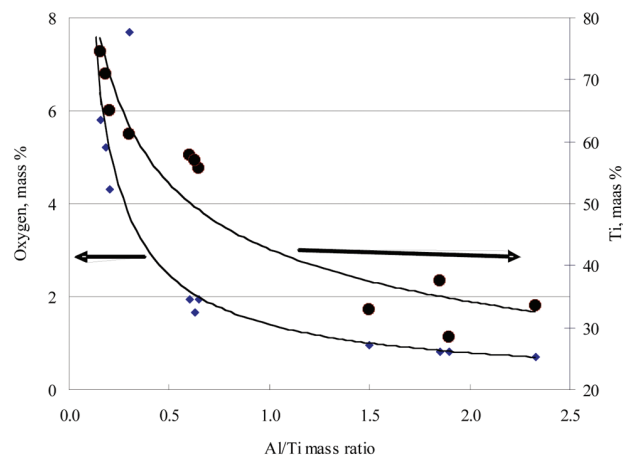*CEN: Comite Europeen de Normalization



**Figure 4—Influence of Al/Ti mass ratio in the feed on oxygen content in the AlTi alloy. Lime addition was adjusted to get a slag containing about 40% CaO**

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*Table III*

**Target slag and alloy compositions, mass per cent**

| Facility | Alloy Composition | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Target | | | | Actual | | | |
| | Al | Ti | C | N | Al | Ti | C | N |
| Sealed | 52.0 | 47.0 | - | - | 34.0 | 58.7 | 0.37 | 0.04 |
| Semi sealed | 52.0 | 47.0 | - | - | 49.7 | 43.8 | 0.25 | 0.23 |
| | Slag Composition | | | | | | | |
| | Target | | | Actual | | | | |
| | CaO | Al$_2$O$_3$ | TiO$_2$ | CaO | Al$_2$O$_3$ | TiO$_2$ | | |
| Sealed | 42.0 | 57.0 | 0.2 | 30.4 | 68.6 | 5.6 | | |
| Semi sealed | 44.3 | 55.5 | 0.2 | 25.8 | 68.6 | 5.8 | | |

order to get an alloy containing 30–80% Ti. The oxygen content varied considerably with the aluminium content in the alloy, possibly as a result of the formation of titanium oxide (TiO), and possibly AlTiO that, partially, reported to the alloy, as suggested by SEM analysis.

### 40 kW testwork

The testwork aimed at producing an alloy containing about 47% Ti (Table III) so as to enhance metal-slag separation by maximizing the density difference between the two phases. Initially, a sealed furnace was employed as a means of preventing air ingress into the facility. Tapping of the furnace products was somewhat difficult, apparently due to the relatively high Al$_2$O$_3$ in the slag as a result of furnace refractory erosion. A semi sealed facility was, therefore, employed in order to allow tapping the furnace by tilting. The feed recipe was the same as before, and a total of roughly 63 kg was processed in four batches.

In both cases, and during good operating conditions, an alloy containing more than 43% Ti (on average) was successfully produced (Table III), although its average aluminium content was somewhat below the target level. The alloy tended to contain both nitrogen (possibly due to air ingress) and carbon (graphite electrode and residual carbon in the lime). The produced slag was relatively high in TiO$_2$. (throughout this paper, the Ti content in the slag is reported as TiO$_2$. It should be noted, however, that the slag contained metallic inclusions and that titanium was present as suboxides such as Ti$_2$O$_3$. As a result, the total slag analysis exceeded 100% in several samples). However, the calculated titanium recovery was well in excess of 88% and reached a high value of 96.2%, based on slag masses and analyses. The tapping difficulties were mostly related to the high Al$_2$O$_3$/CaO ratio in the slag, which exceeded the target as a result of refractory erosion and, possibly, oxidation of the aluminium in the feed. These difficulties could have been influenced by the presence of nitrogen and carbon in the resulting alloy, either as carbides, nitrides, oxycarbides, and/or oxynitrides. Such compounds exhibit very high melting points, and could have settled out and accumulated in the bottom of the furnace as a solid layer. Nevertheless, the results were

encouraging enough to warrant undertaking a prolonged campaign in order to produce sufficient alloy for testing as a grain refiner, and to demonstrate the process at a larger scale.

### 200 kW testwork

In this phase of the development work, several metallurgical and operational parameters were investigated, as shown in Table IV. Initially, a water cooled shell was employed to continuously produce an alloy containing 42% Ti and 55.8% Al, (feed composition, 34.0% TiO$_2$, 42.5% aluminium, and 23.4% lime). Freezing of the bath, particularly nearby the side walls and the hearth, made it difficult to tap the products by drilling through the taphole. Therefore, oxygen lancing was relied on to recover the products. In addition to lower titanium content, the tapped alloy tended to contain relatively high levels of carbon, nitrogen and oxygen, although the facility was purged with argon at 40–50 ℓ/min. On the other-hand, the dig out metal tended to be richer in titanium. The major metallic impurities include silicon and iron. Notice that carbon and nitrogen contents in the dig-out metal were generally higher than those of the tapped alloy (Table V). In the meantime, the average oxygen content in the tapped metal was marginally higher than that in the dig-out alloy, possibly due to oxidation as a result of oxygen lancing.

In the second condition, water cooling to the shell was turned off and aluminium addition was increased in order to produce an alloy containing 35–38% Ti and 60–63% Al (feed composition, 34.2% TiO$_2$, 47.7% aluminium, and 17.2% lime). Tapping was accomplished through the upper taphole by tilting. The tapped alloy analysed about 25% Ti, on average, where the metal recovery was as high as 99% (based on slag masses and analyses). The contaminants were mostly, silicon and iron. Three batches were processed totalling about 340 kg before a build-up was experienced inside the furnace.

After flushing the furnace with a few batches of mild steel, and then a mixture of lime and aluminium, and tapping through the bottom taphole, Condition 3 was attempted. Here, lime addition was adjusted in order to produce a slag with

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35% CaO. Extremely low titanium analysis in the alloy led to changing to sequential feeding in the next two conditions. This mode of feeding called for melting a mixture of 55.8% $TiO_2$, 15.7% lime, and 28.5% alumina, followed by charging a mixture of 75.2% Al and 24.8% lime so that the target slag and metal compositions would be as per Condition 3. Good success was achieved where, in each condition more than 8 batches were smelted before a serious build up was experienced. In addition, the produced alloy contained relatively higher titanium in comparison to Conditions 1 and 3 on a more consistent basis. In particular, sequential feeding with bottom taphole lancing (Condition 5) resulted in an alloy containing 38% Ti in a few taps, and a slag analysing about 1.5% $TiO_2$. Although lancing led to iron contamination, certain alloy samples contained less than 1% Fe.

Co feeding and tapping by lancing through the bottom taphole (Condition 6, with similar feed composition to that of Condition 3) significantly improved the alloy quality in terms of reaching relatively high titanium content throughout most of the taps. In addition, the slag was relatively low in titanium where a low value of roughly 3.0% $TiO_2$ was attained. The improved operation, and relatively good metallurgical results obtained in the last two conditions, warranted the partial and gradual substitution of pigment grade $TiO_2$ with rutile. The main objective here was to investigate the zirconium behaviour and its influence on the quality of the alloy.

On average, and with 25% rutile and 75% $TiO_2$ (Condition 7), the produced alloy was of similar composition to that produced in Condition 6. The zirconium content in the

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*Table IV*

**Experimental conditions and selected results**

| Condition no. | Operational and metallurgical targets | Mass fed, kg | Ti in the alloy, mass % | $TiO_2$ in the slag, mass % |
|---|---|---|---|---|
| 1 | Co-feeding, water cooled shell, tapping by drilling through lower taphole, 40% CaO in slag, 42% Ti in the alloy | 795 | Tapped: 4.5-11.8 Digout: 29.6-59.1 | Average: 2.51 Best: 1.01 |
| 2 | Co-feeding, air cooled shell, upper taphole tilting, 40% CaO in slag, 38% Ti in the alloy | 352 | Average: 24.6 Best: 27.0 | Average: 1.57 Best: 0.15 |
| 3 | As in 2, 35% CaO in the slag | 524 | Average: 0.1 Best: 0.2 | Average: 13. Best: 0.13 |
| 4 | As in 3, sequential feeding | 1000 | Average: 7.9 Best: 11.3 | Average: 8.7 Best: 3.13 |
| 5 | As in 4, tapping by lancing through lower taphole | 830 | Average: 13.3 Best: 38.0 | Average: 5.7 Best: 1.5 |
| 6 | As in 3, tapping by lancing through lower taphole | 800 | Average: 31.1 Best: 39.4 | Average: 4.9 Best: 3.0 |
| 7 | As in 6, 25% rutile and 75% $TiO_2$ | 738 | Average: 31.4 Best: 42.9 | Average: 4.6 Best: 1.6 |
| 8 | As in 6, 50% rutile, and 50% $TiO_2$ | 1025 | Average: 24.4 Best: 31.2 | Average: 6.1 Best: 3.5 |
| 9 | As in 6, 75% rutile and 25% $TiO_2$ | 911 | Average: 33.3 Best: 35.8 | Average: 7.3 Best: 1.5 |

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*Table V*

**Weighted average chemical analyses of the alloy, mass per cent**

| Condition | Ti | Al | Si | Fe | Zr | N | O | C |
|---|---|---|---|---|---|---|---|---|
| 1 | 8.2 (44.3) | 15.2 (9.8) | 8.6 (14.6) | 21.1 (22.7) | <0.05 | 0.04 (3.11) | 1.24 (1.02) | 0.19 (0.45) |
| 2 | 24.61 | 60.56 | 3.42 | 3.04 | <0.05 | NA | NA | NA |
| 3 | 0.09 | 82.47 | 0.38 | 3.73 | <0.05 | NA | NA | NA |
| 4 | 7.94 | 76.37 | 0.50 | 4.07 | <0.05 | NA | NA | NA |
| 5 | 13.33 | 70.53 | 0.37 | 7.92 | <0.05 | NA | NA | NA |
| 6 | 31.08 | 56.79 | 1.05 | 4.62 | <0.05 | NA | NA | NA |
| 7 | 31.40 | 54.51 | 1.16 | 5.47 | 0.09 | NA | NA | NA |
| 8 | 24.44 | 61.93 | 0.98 | 5.72 | 0.12 | 0.01 | 0.28 | 0.05 |
| 9 | 28.39 | 49.97 | 1.07 | 10.32 | 0.17 | NA | NA | NA |

Values in parenthesis refer to dig-out metal. NA: Not Analyzed for

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alloy was 0.13% and lower. With 50% rutile, (Condition 8), the alloy tended to analyse less titanium while the zirconium analysis remained essentially uncharged, on average. Further increase in rutile addition to 75% (Condition 9) yielded an alloy with about 33.3% and 0.15% Zr, on average.

## Discussion

DC arc production of 30–40% Ti-Al alloy was demonstrated to a large extent at 40 and then 200 kW scale of operation. The results obtained during good operating periods tended to confirm the laboratory scale data in terms of titanium content in the alloy and its recovery. Certain operational and equipment challenges, however, were experienced and contributed to the contamination of the alloy particularly with regards to iron, silicon, carbon, nitrogen and oxygen. In total, about 7 tons of raw materials were smelted, producing about 3.5 tons of high TiAl alloy.

As stated earlier, the testwork relied on argon purge in order to prevent air ingress into the facility. This approach was not effective enough and led to the partial oxidation of aluminium and the produced alloy, particularly at the initial stages of the reduction process where the density of the alloy is lower than that of the slag. In addition, air ingress appeared to have caused the formation of nitrides and oxy-nitrides of aluminium and titanium[4].

With upper taphole tapping (Conditions 1-4), the tapped alloy tended to analyze lower Ti, compared to the target. This is believed to be related to the formation of a relatively light alloy phase that remained on top of the slag. In the meantime, the dig out alloy was, generally, richer in titanium and therefore, of higher density than the slag, allowing it to settle out and form a metal layer in the bottom of the furnace. Settling of this Ti rich alloy phase is believed to have been enhanced by the presence of carbides, nitrides, as well as heavy elements including iron, zirconium, etc.

The data obtained with sequential feeding and upper taphole tapping (Conditions 4) suggests that the kinetics of the reduction process may have been limited by the diffusion of TiO$_2$ into the slag-metal interface, resulting in relatively high TiO$_2$ in the slag and low Ti content in the metal. Improvements were realized when the bottom taphole was utilized for tapping (lower average bath depth), which facilitated the prompt removal of both the alloy and slag, and thereby minimized the diffusion paths to a certain extent .

Similar results were achieved with co-feeding and tapping through the lower taphole (Conditions 6–9). In these conditions, the titanium analysis in the alloy approached the target value in several taps and averaged between 24.4 and 33.3%, suggesting that titanium recovery to the alloy phase was 87% on average, and could have exceeded 95%.

Zirconium analysis in the alloy obtained in the last three conditions remained very low and within acceptable levels when compared with the results of the lab scale tests. This might be due to the volatilization of zirconium as ZrO(g), particularly in the arc attachment zone where the temperature could be well above 2500°C.

As shown in Table V, the major impurities in the alloy are iron, silicon, carbon, nitrogen and oxygen. Oxygen lancing contributed significantly to the iron levels, in addition to the use of mild steel to flush the furnace build-up when it occurred, and the presence of iron in the lime and rutile. Silicon and carbon levels can be controlled by proper selection of the raw materials (lime and rutile). However, oxygen and nitrogen may only be limited by a proper design of the facility whereby air ingress can be eliminated.

Incomplete metal slag separation was experienced in various conditions and lead to 5 to 15% metal losses to the slag[5]. Consequently, the calculated titanium recovery and accountability were relatively low at 87–96% and 88–92%, respectively. This difficulty can be overcome, to a large extent, by bath stirring and by small addition of fluorspar and/or cryolite.

## Conclusions

Laboratory scale testwork showed that a 30–40% TiAl alloy was produced at 1600–1625°C, using pure TiO$_2$, lime and aluminium. Titanium recovery was very high reaching more than 99%. The levels of major impurity elements in the alloy were low and within acceptable levels.

Large scale production of the alloy was undertaken at the 40 and then 200 kW DC arc furnaces, whereby an alloy containing 24–35% Ti was tapped in several batches. Titanium recovery to the alloy phase exceeded 87% and could have been as high as 95%. The use of rutile in the feed recipe resulted in an alloy containing less than 0.2% Zr when rutile to TiO$_2$ mass ratio in the feed was 3-to-1.

Further optimization work is still required in order to adequately demonstrate the process. The work could aim at minimizing iron contamination upon tapping, and the prevention of carbon and nitrogen pick up by the alloy.

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