RECOVERY OF VANADIUM AND NICKEL FROM PETROLEUM FLYASH

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Key words: Flyash, DC arc furnace, Pre-treatment, Smelting, Vanadium, Nickel, Extraction, Recovery.

ABSTRACT

Flyash is a powdery residue generated by the power stations that use heavy oil as the source of fuel. The ash poses a threat to the environment due to the presence of certain heavy elements such as chromium. At the same time, it contains valuable metals, including vanadium and nickel, that are extractable if an economical and environmentally acceptable process can be developed. To this end, Mintek, in co-operation with Oxbow Carbon and Minerals, undertook a major research program over a two-year period in an attempt to find the most suitable technology to deal with this ash. After considering various alternatives, a process flowsheet was developed and used as a basis to identify the testwork required for the major processing units. The flowsheet consisted of a drying stage at moderate temperatures of about 150 ^{0}C, a de-carburization and de-sulphurization (pre-treatment) stage, and finally a smelting step in a DC arc furnace.

Testwork carried out at the 40 kW scale, using pre-treated flyash, indicated the feasibility of producing a ferrovanadium alloy containing more than 15 per cent vanadium and 6 per cent nickel, with vanadium recovery of more than 89 per cent, and a disposable slag based on USA EPA safe disposal criteria. Several parameters were studied in order to optimise the smelting stage including operating temperature, aluminium addition, the use of ferrosilicon as the reducing agent and lime (flux) addition. The results of the investigation are presented in this paper.

INTRODUCTION

The power generation industry that uses heavy oil (Venezuelan oil for example) produces a solid residue called flyash that contains a relatively high heavy metal content, particularly vanadium oxide (typically 3% V_2O_5) and nickel oxide (about 1%NiO). In addition, and in order to meet strict NO_x specifications during burning, the residual carbon level in the flyash is very high (typically 80% on dry basis). The produced flyash is currently land-filled in lined ponds. Water is used in most cases to slurry the material to the disposal site. In certain instances, coal ash has been mixed with oil ash resulting in the dilution of the metal oxides in that final ash.

There are increasing environmental concerns regarding the possible leaching and seepage of heavy metals into ground water adjacent to the storage ponds. Therefore, Western countries are considering new legislation, which would classify the flyash as a hazardous material. This would lead to a considerable increase in the costs associated with storing the flyash. However, a relatively high level of vanadium, and possibly also the nickel,

could make the oil flyash a source of these metals, provided that a suitable and applicable technology is developed to treat it.

Ferrovanadium alloys are produced commercially by the reduction of vanadium ore, slag, or technical grade vanadium oxide with ferrosilicon, aluminium and to a lesser extent by carbon, Milleron (1983) and Moore (1992). A charge consisting of vanadium oxide, reductant, fluxing agents, etc, is fed to an electric arc furnace that is maintained at temperatures of about $1600^{O}C$ and higher, yielding either a 40% V alloy or an 80% V alloy. Depending on the nature and therefore the exothermicity of the reaction, a thermite reactor may be used as an alternative to an electric arc furnace.

The use of plasma reactors to produce FeV alloys from vanadium oxides was studied by MacRae et al. (1976) and by Minura and Taniuchi (1977). Processing of vanadium-containing flyash was investigated by Pickles and Alcock (1983). An extended arc flash reactor was employed to treat a flyash containing about 8% NiO and 42% V_2O_5. Anthracite and petroleum coke were used as reducing agents. A FeNi alloy was first produced by selective reduction of the ash, followed by final reduction to a 43 % V alloy. After thorough evaluation and consideration, Mintek developed a generalised process flowsheet that consisted of a pre-treatment stage, namely drying or de-watering of the flyash followed by a combustion step to remove carbon and sulphur. The treated ash is then reduced by smelting in a DC arc furnace, using ferrosilicon or aluminium, to produce a vanadium alloy and a disposable slag. A testwork program was designed to demonstrate the application of this flowsheet to the ash. The test program covered 60 kVA induction furnace tests, and a 40 kW DC arc furnace tests. The results of the investigation are presented in the following sections.

EXPERIMENTAL EQUIPMENT AND PROCEDURES

The testwork was conducted in two stages. The first stage comprised exploratory tests in a 60kVA induction furnace in order to evaluate the influence of certain parameters on the quality of the ferrovanadium alloy produced. This work constituted the basis for the next stage that consisted of 40 kW DC arc furnace smelting tests. The work was preceded by drying, de-carburizing and de-sulfurizing (pre-treatment) of the as-received flyash. This pre-treatment stage was necessary in order to prepare a suitable material for the smelting tests, such that the ferro-alloy produced would contain as little carbon and sulphur as possible so that when used as a steel additive, the levels of these two elements would not exceed the specified maximum standard. As shown in Table 1, the as-received flyash contained about 33% moisture, 33% carbon and 2.5% V_2O_5. The ash was first pre-treated in rectangular steel trays (200x100x30mm) in a small muffle furnace to remove the moisture and most of sulphur and carbon. The pre-treatment conditions were: temperature = $1000^{o}C$, time = 2 - 24 hours, atmosphere = air, sample size = 350g, and sample depth = 20mm. These tests had two objectives: the first being to determine the pre-treatment conditions necessary to remove sulphur and carbon, and secondly to prepare a small sample (about 1.5kg) for the induction furnace tests. Most of the contained carbon and sulphur was removed in about 4 hours, producing a pre-treated ash containing about 0.012%C and less than 0.01%S. The pre-treated ash was then screened to -1mm in order to remove most of Al_2O_3-SiO_2 beads that were observed to be present in the ash. The fine fraction (-1mm) was used for the induction furnace tests. The chemical assay of the ash is given in Table 1.

The induction furnace (Figure 1) consisted of a medium frequency power supply, water-cooled copper coils, a graphite susceptor and an alumina tube located between the induction coils and the graphite susceptor. An alumina or graphite mould was placed on

the base of the susceptor and acted as a housing for three A5 re-crystallized alumina crucibles. The crucibles were arranged in a triangular position with respect to each other. A B type thermocouple was used to monitor, control and record the temperature at the central space between the crucibles. The water-cooled lid of the furnace contained three entry ports respectively for argon purge, a thermocouple, and for off-gas extraction.

The induction furnace tests were carried out as follows: a 60g sample of the pre-treated ash was placed in each of the three crucibles along with a given amount of flux (Table 2). The crucibles were then placed inside a graphite holder and the furnace power was switched on. The power input was increased gradually until a temperature of $1600^{o}C$ was reached. This operating temperature usually required a power input of about 45 kW.

During this heating period (3 hours), argon was blown into the furnace at a rate of $3l$/min to provide an inert atmosphere (by minimising air ingress) during the smelting period. Once the desired temperature was reached, the power was turned off and the reductant (either ferrosilicon or aluminium) was added to each crucible through an alumina feed pipe inserted through the argon purge port. The power was then turned on and adjusted regularly to maintain a temperature of $1600^{o}C$. After a holding period of 2 hours, the power was switched off and the crucibles were allowed to cool down overnight. Clean metal and slag samples were then taken, pulverised and chemically analysed.

The experimental conditions employed, shown in Table 2, aimed at investigating the influence of lime addition, nature and amount of reductant, and other fluxing agents on the smelting process.

Upon the conclusion of these tests, and the evaluation of the results, a decision was taken to proceed with the 40 kW DC arc furnace tests. The facility consisted of a water-cooled steel shell, a feed system, a 100kVA power supply and a gas extraction system (Figure 2). The steel shell was lined with high MgO rammable, while the roof was lined with high alumina castable. A graphite block was placed in the bottom of the furnace and was connected to a steel anode pin, which was attached to the anode cables. The furnace roof contained a central entry port for the solid graphite electrode, which acted as the cathode, and two additional ports for feeding raw materials, and for gas extraction, respectively. The gas handling system comprised an off-gas pipe and bag plant for gas cleaning. Due to the fineness of the pretreated flyash, the furnace feed was manually charged into the furnace through the feed port.

The as-received flyash was dried, de-carburized and de-sulphurized in a large muffle furnace. This furnace consisted of a steel housing, refractory insulation and heating elements located at the inner sidewalls. A temperature controller was used to maintain the furnace temperature at $1000^{o}C$. The flyash was placed in steel trays with a depth of about 50mm. The pre-treatment lasted for about 16 hours to ensure acceptable levels of carbon and sulphur, as with the induction furnace tests. The pre-treated flyash (about 150kg) was then screened to –1mm to remove the Al_2O_3-SiO_2 beads. Lime to be used as a flux was calcined at $1000^{o}C$ for 16 hours in the same facility to remove any moisture present as well as carbon dioxide.

The smelting tests were carried out over a three-day period in a 100 kVA DC arc facility. The tests were carried out as follows: the arc was established between the cathode and the graphite block at power levels of 15-25kW. This was followed by feeding of the pre-mixed raw materials (Taps 1-6 only), as shown in Table 3, over a period of 1 to 1.25 hours (about 10kg/h).

The power to the furnace was then decreased to a level corresponding to the measured energy losses for an additional 10 minutes to allow additional time for the completion of the reactions, and to accomplish better slag-metal separation. The furnace was then

tapped, and slag and metal samples were taken and chemically analysed. Quenched slag samples were also prepared for leachability tests.

In the latter taps, a smelting procedure of sequential pre-melting and reduction steps was adopted, as opposed to the single-step smelting procedure of the earlier taps. Specifically, during taps 7 to 11, a mixture of flyash and lime was pre-melted in the furnace over a period of 45 minutes (pre-melting), followed by subsequent addition of the reductant over the next 15 to 25 minutes (sequential feeding).

EXPERIMENTAL RESULTS AND ANALYSIS

The main results of the induction furnace tests clearly proved the feasibility of producing a crude ferrovanadium alloy from the pre-treated flyash. In general, aluminium addition of 14 to 18% (mass per cent based on mass of flyash) gave better results than ferrosilicon reduction in terms of lower V_2O_5 content in the slag (down to about 0.7%), and higher vanadium analyses in the metal (up to 18.0%V). In comparison ferrosilicon addition of 17.5 to 22.5% resulted in a V_2O_5 in the slag of at least 3.4%, and the alloy contained between 7.8 and 18.0%V. The addition of fluxing agents such as Na_2O, K_2O, CaF_2 did not appear to have improved the extractions significantly. Also, the effect of lime addition on both metal grade and slag analysis was inconclusive. Therefore, further 100 kVA tests were conducted with a few changes in the feed mix, as shown in Table 3.

Full chemical analyses of slag and metal samples appear in Table 4 and 5 for all taps conducted, along with their corresponding masses. Figure 3 suggests that increased ferrosilicon addition beyond 25% does not show any lower vanadium losses to the slag, or increased V-content in the metal. In fact, the vanadium content in the metal dropped from about 6.5% with 25% FeSi addition to about 5.5% with 30% FeSi addition. This trend could be attributed to the inability of the slag to absorb more silica, resulting in a silica-saturated slag. As a result, any additional ferrosilicon would remain as metal, leading to a higher Si-content and a lower V-content in the produced alloy. Overall, the test results indicate that the maximum vanadium analysis in the alloy produced was about 6.7% together with a slag containing about 2% V_2O_5. Another reason for the high V_2O_5 in the slag could be the mechanism of ferrosilicon reduction of vanadium oxide. A ferrosilicon particle, solid or liquid, being of similar density to that of the slag, begins to react on the slag surface depleting it in silicon. As the reaction progresses with time the particle becomes denser. This continues until its density becomes higher than that of the slag, where it starts to percolate and coalesce with other particles until it reaches the metal layer and dissolves. During its descent, the FeSi particle continues to react with the slag, provided that the surrounding slag is not saturated with silica and the silicon content of the metal is higher than the equilibrium value. Deviation from equilibrium is the result of mixing (turbulence) within the bath, and the particle descent time is shorter than the time required for the reaction to proceed to equilibrium.

In contrast, increased aluminium addition continued to lower the V_2O_5 content in the slag (Figure 4), where the V_2O_5 content dropped to about 0.68% with 19% Al addition. The produced alloy contained about 14.6 to 15.1% V when 14 to 20% Al was used, although it appeared that further Al-addition beyond 14% did not improve the metal grade. This is an indication that silica reduction by aluminium becomes significant, particularly with higher Al-addition and proportionally less lime addition. The reduction of V_2O_5 with aluminium takes place mostly at the bath surface, as the slag density remains higher than that of aluminium throughout the progression of the reactions. This requires that vanadium oxide diffuses from the bulk of the slag towards the reaction site at the slag surface, the rate of which will depend on several factors including temperature,

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concentration gradient, slag fluidity, etc. Another aspect of aluminium reduction is its reaction with silica in the slag once the slag surface becomes depleted with more readily reducible metal oxides. The produced silicon metal descends through the slag and to some extent reacts with other metal oxides before it reaches the metal layer. This is evident in the Si-content in the metal where there did not seem to be any significant difference between the alloy silicon content whether aluminium or ferrosilicon was used as a reducing agent during the testwork.

The importance of lime addition can be seen in Figure 5. These tests were carried out with about 14% Al addition. With sequential feeding, the V_2O_5 content in the slag dropped from 1.64% with 15% CaO addition, to 1.12% with 25% CaO addition. At the same time the vanadium analysis in the metal increased from 9.85 to over 14%. Co-feeding of the reactants with 20% lime addition resulted in a slag higher in V_2O_5 content, but the metal grade was relatively high at about 14.5%. Increased lime addition is believed to contribute to the extent of reduction of the metal oxides by lowering the activity of silica or alumina such that the slag capacity to absorb these components is increased. Another positive aspect of increased lime addition is its influence on reducing the slag viscosity, contributing to a better slag/metal separation. The latter effect could have been responsible for the higher vanadium content in the metal and its improved recovery with increased lime additions as will be shown later.

Vanadium and nickel recoveries to the alloy phase were calculated based upon: i) slag masses and analyses (V-slag, Ni-slag), and ii) metal masses and analyses (V-metal, Ni-metal). Generally, the calculated recoveries were lower when based on metal masses and analyses. This could be due to the entrained metallic droplets in the slag and because of the difficulty of separating the two phases, leading to a lower metal mass and a higher slag mass and analyses for V_2O_5, NiO, FeO etc. Nevertheless Figures 6 to 8 suggest that the nickel recovery was always high reaching more than 98% (based on slag analysis), regardless of the experimental conditions employed. As far as the vanadium recovery is concerned, increased aluminium addition appears to be very important, (Figure 6) where the recovery increased from about 20% with 6% Al addition to about 90% with 19% Al addition. However, increased ferrosilicon addition in the range of 20 to 30% did not seem to improve vanadium recovery at all (Figure 7), while lime addition increased it moderately up to 20% CaO, followed by a sharp increase to 85% at 25% CaO addition (Figure 8).

TCLP

Water-cooled (quenched) slag samples were subjected to leachability testing using the USA EPA TCLP procedure. A sample from the pre-treated flyash was also tested using the same procedure. The results are shown in Table 6, along with the stated permissible maximum. Clearly all slag samples passed the leachability tests, indicating that the slag can be safely disposed of.

MASS BALANCE

The overall mass balance shows an overall accountability of about 103% (Table 7). This is mainly due to refractory pick-up by the slag, as a result of chemical erosion and thermal degradation (about 3.7kg), and to iron dissolution from the anode (about 1.1kg). Taking these figures into account, the overall accountability approaches 100%.

Individual accountabilities of major elements are shown in Table 8. Magnesium and iron accountabilities are high at about 157% and 122% respectively, confirming that anode

and refractory erosion contributed to the high overall accountability. Accountabilities of other elements range from 87.5% for vanadium to 99.8% for manganese. It is believed that metallic inclusions in the slag may have contributed to low accountabilities for elements such as V, Ni and Si, while calcium and aluminium accountabilities may have been influenced by slag being soaked into the furnace refractory lining.

ENERGY BALANCE

In the presence of ferrosilicon as reducing agent, the furnace energy input ranged from 20.5 to 26.2kW. The rate of energy losses were measured at about 19.2kW, resulting in a thermal efficiency of about 23 to 27%, with a slag tapping temperature of about 1600 to 1630°C. Energy requirement for the process therefore was estimated at 0.5 to 0.6kWh/kg feed (excluding energy losses).

With aluminium reduction, energy input ranged from 22.9 to 38.4kW, while the measured energy losses were 17.8 to 34.0kW, giving a slag tapping temperature of about 1550 to 1700°C. Energy requirement for the process, therefore, ranged from 0.37 to 0.43kWh/kg feed.

CONCLUSIONS

Oil flyash was successfully de-carburised and de-sulphurised at 1000°C, giving a pre-treated ash suitable for smelting in a DC arc furnace for the purpose of producing a ferrovanadium alloy containing up to 15%V and 6%Ni. The results indicated that utilizing aluminium as the reducing agent was more effective than ferrosilicon in terms of both metal extraction and alloy quality. Varying the lime addition appeared to improve vanadium extraction with both ferrosilicon and aluminium as reductants. However, with silicon reduction, more lime was required to neutralise the additional silica in the slag.

RECOMMENDATIONS

The encouraging results obtained from the testwork suggested that aluminium reduction should be pursued, rather than ferrosilicon, at a scale of 200kW or higher. This would give enough operational and metallurgical data required for the scale-up to a commercial installation of 1 to 2MW. Any future testwork could also investigate the possibility of selective reduction of nickel (to produce a FeNi alloy) followed by vanadium extraction to make a FeV rich alloy. Finally, it is recommended that an alternative pre-treatment process be investigated with the objective of utilizing the contained carbon in the process.

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Table 1. Chemical analyses of lime, FeSi, Al and flyash before and after treatment, mass percent

Component	Lime	Flyash		Component	Al	FeSi
		Before pre-treatment	After pre-treatment			
V_2O_5	NA	2.45	7.18	Al	98.00	0.10
SiO_2	0.35	15.4	37.6	Fe	0.71	25.60
Fe_2O_3	0.21	3.68	11.78	Si	0.32	73.41
CaO	97.3	1.35	3.04	Mn	0.25	0.15
MgO	0.64	3.65	8.41	Cr	0.02	0.05
MnO	1.0	0.04	0.09	Ni	0.02	0.05
Al_2O_3	0.21	8.59	21.45	Co	0.02	0.05
TiO_2	NA	0.45	0.98	C	0.02	0.18
NiO	NA	0.71	1.88	Mg	0.45	0.00
Cr_2O_3	NA	0.30	0.62			
S	NA	0.91	0.28			
C	NA	33.0	0.03			
K_2O	NA	0.81	2.31			
Na_2O	NA	0.43	0.66			
H_2O	NA	33.4	0.01			

NA: not analysed.

Table 2. Feed recipes for the induction furnace tests

Test No	Lime, g	FeSi, g	Al, g	Other fluxes, g
1	18	12.0	0	0
2	18	10.5	0	0
3	18	13.5	0	0
4	12	0	9.6	0
5	12	0	8.4	0
6	12	0	10.8	0
7	9	0	9.6	0
8	15	0	9.6	0
9	21	12.0	0	0
10	15	12.0	0	0
11	12	0	9.6	CaF_2 = 1.2g
12	12	0	9.6	CaF_2 = 2.4g
13	12	0	9.6	Na_2O = 1.2g
14	12	0	9.6	K_2O = 1.2g
15	12	0	9.6	0
16	12	0	3.4	0

Table 3. Experimental conditions of the 100kVA DC arc furnace tests

Test No	Al, kg	FeSi, kg	Lime, kg
1	0	1.6	2.4
2	0	1.6	2.4
3	0	2.0	2.4
4	0	2.4	2.4
5	1.3	0.0	1.6
6	1.1	0.0	1.6
7	1.5	0.0	1.6
8	1.3	0.0	1.2
9	1.3	0.0	2.0
10	0	2.0	2.8
11	0.45	0	1.0

Table 4. Chemical analyses and masses of slag produced in the 100 kVA DC arc tests

Tap No.	Mass kg	MgO	SiO_2	FeO	Al_2O_3	NiO	CaO	MnO	V_2O_5	TiO_2
1	9.15	8.93	39.0	5.51	17.3	0.46	22.60	0.31	3.11	0.68
2	10.32	10.30	38.4	4.03	17.4	0.10	23.00	0.17	2.00	0.49
3	10.13	9.71	37.9	3.72	17.9	0.05	23.34	0.17	3.08	0.49
4	10.60	9.36	38.7	3.47	17.8	0.11	23.30	0.16	3.04	0.47
5	7.28	8.97	25.4	0.67	40.5	0.03	17.60	0.18	1.83	0.51
6	9.08	9.11	26.5	0.73	38.2	0.03	17.70	0.17	2.14	0.59
7	9.57	17.30	20.7	0.39	41.2	0.03	16.40	0.17	0.68	0.36
8	5.14	10.80	21.4	0.65	45.3	0.30	15.05	0.13	1.64	0.46
9	11.90	16.30	20.1	0.21	40.2	0.03	17.70	0.09	1.12	0.41
10	11.28	11.00	32.5	6.20	18.7	0.03	24.10	0.22	3.60	0.52
11	9.92	10.10	31.1	6.70	25.4	0.03	17.00	0.24	4.60	0.75

Table 5. Chemical analyses and mass of metal produced in the 100 kVA DC arc tests, mass per cent

Tap No.	Kg	Si	Fe	Al	Ti	Mn	V	Ni
1	2.345	25.5	61.5	0.05	0.44	0.37	5.46	4.05
2	2.030	33.2	49.7	0.01	0.92	0.70	6.69	5.70
3	2.226	40.9	43.2	0.05	0.89	0.60	6.46	5.12
4	2.554	44.4	41.6	0.05	0.85	0.55	5.66	4.26
5	1.184	21.8	50.4	0.05	1.17	0.51	14.60	7.86
6	1.182	18.8	51.7	0.05	1.01	0.59	15.10	9.17
7	1.288	31.2	38.9	0.12	1.59	0.97	14.85	6.25
8	1.544	23.9	58.6	0.05	1.01	0.64	9.85	4.04
9	1.937	23.8	49.4	0.05	1.21	0.94	14.10	6.21
10	2.144	47.5	36.5	0.05	0.75	0.49	4.01	6.27
11	0.418	13.9	54.0	0.05	0.30	0.12	8.56	17.70

Table 6. TCLP results: concentrations of various elements, ppm

Element	Max	Tap 5	Tap 6	Tap 7	Tap 8	OFA
Cr	5	0.160	0.170	0.180	0.270	0.800
As	5	<0.002	<0.002	<0.002	<0.002	<0.002
Se	1	0.030	0.010	<0.002	<0.002	<0.002
Ag	5	<0.002	<0.002	<0.002	<0.002	0.270
Cd	1	<0.002	<0.002	<0.002	<0.002	<0.002
Ba	100	0.225	0.130	1.200	0.410	0.165
Hg	0.2	<0.001	<0.001	<0.001	<0.001	<0.001
Pb	5	0.050	0.010	0.002	0.010	<1.00

OFA: pre-treated oil flyash

Table 7 Overall Mass Balance

Materials in, kg		Materials out, kg	
Oil Flyash	88.0	Metal	18.85
Ferrosilicon	9.6	Slag	104.37
Aluminium	6.95	Fume	1.40
Lime	22.0	Dig-out Slag	6.98
Total	126.55	Total	131.60
Accountability	103.99	Unaccounted for, kg:	-5.05
Refractory Pick-Up	3.67		
Iron Pick-Up (anode)	1.12		
Corrected Accountability. %	100.21	Unaccounted for, kg	-0.26

Table 8 Overall Elemental Accountabilities

Element	Accountability, per cent
Mg	156.9
Si	93.1
Fe	121.8
Al	91.9
Ni	91.1
Ca	87.5
Mn	99.8
V	87.5
Ti	112.6
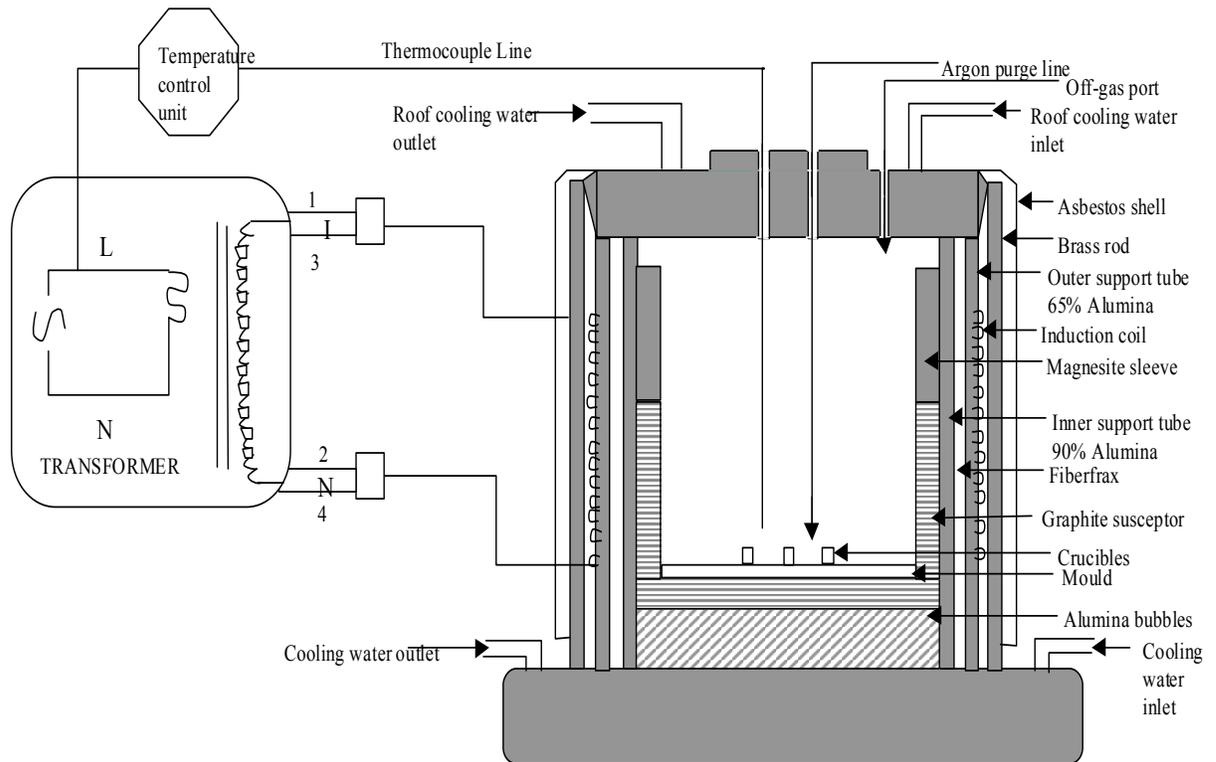
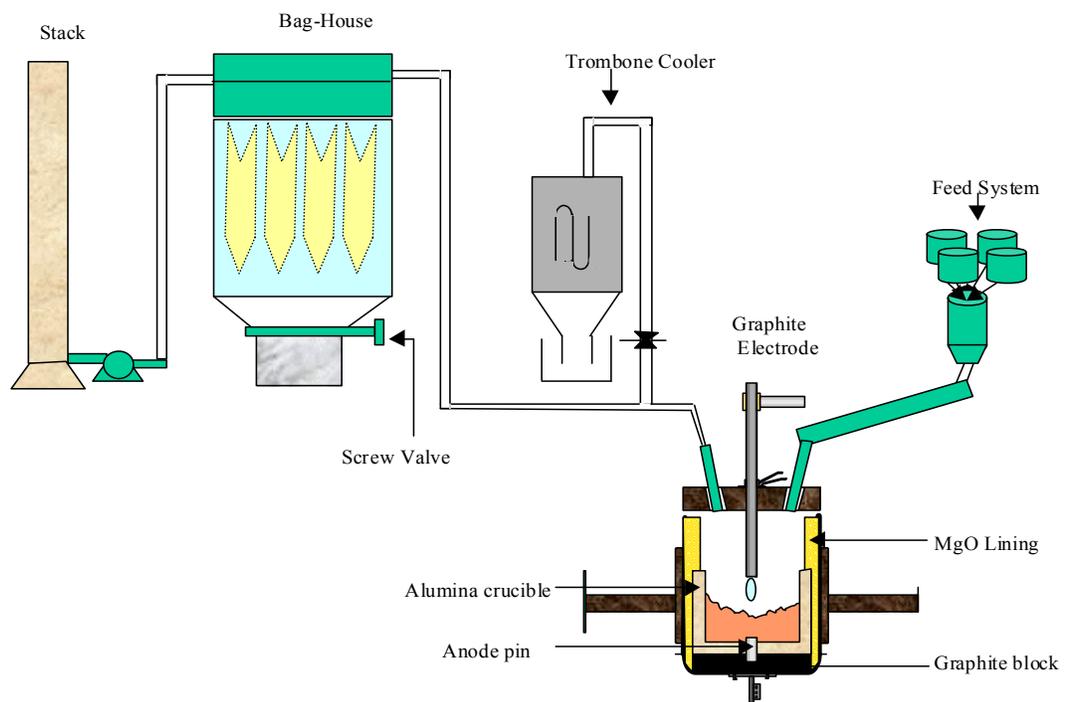Figure 1. Schematic of the 60 kVA induction furnace.

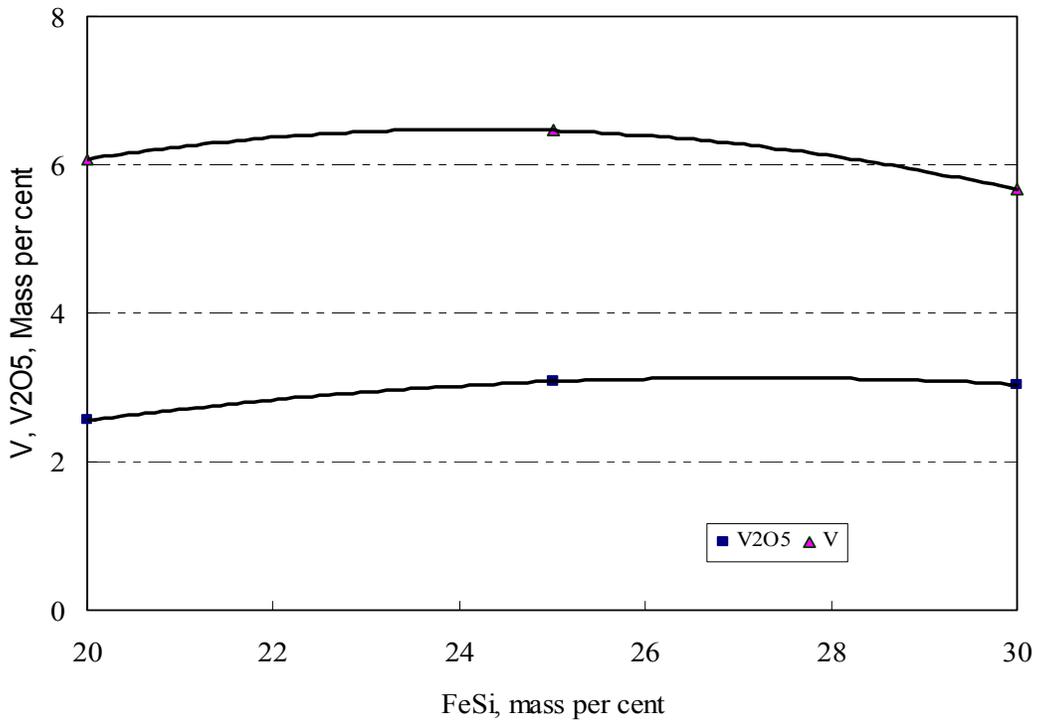Figure 2. Layout of the 100 kVA dc arc facility

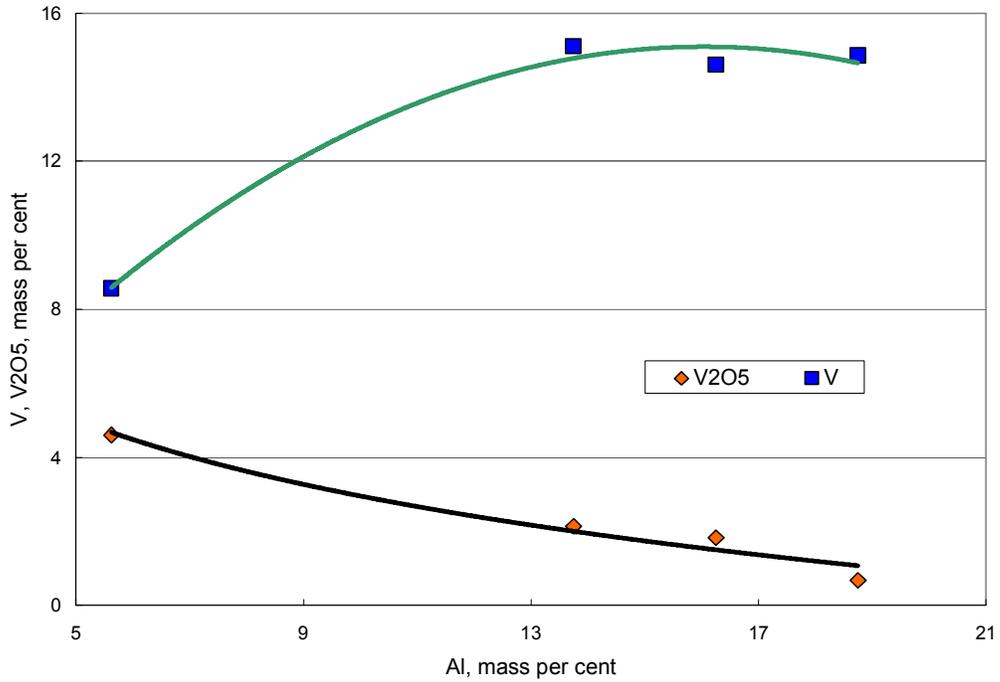Figure 3. Influence of FeSi addition on V_2O_5 in the slag and the V-content in the metal.

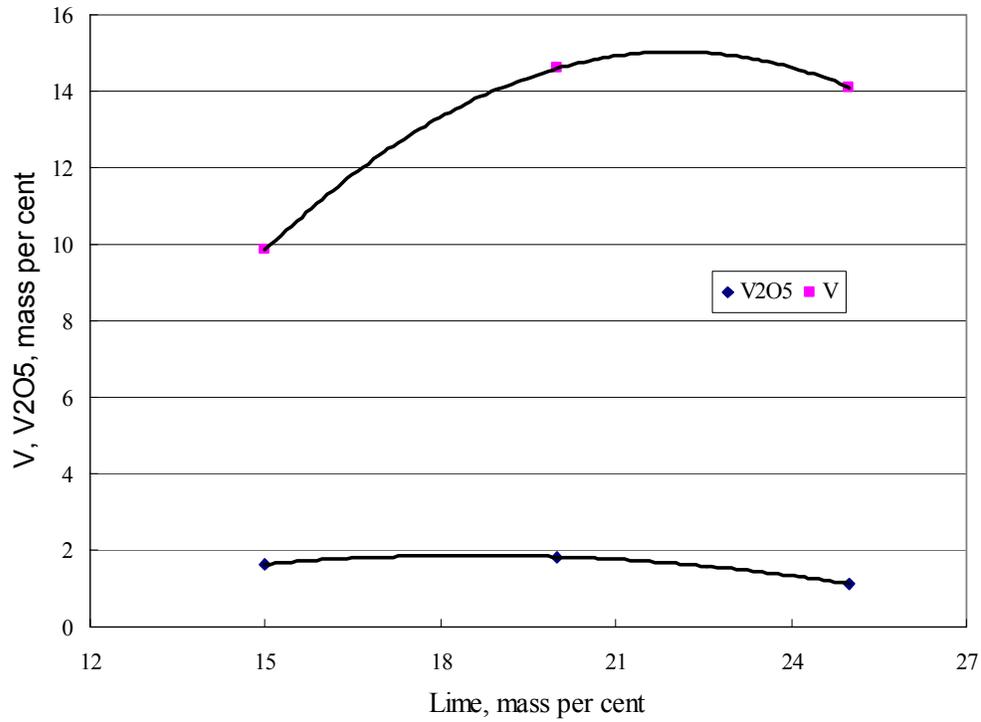Figure 4. V_2O_5 analysis in the slag and V-content in the metal versus aluminium addition.

Figure 5. V$_2$O$_5$ analysis in the slag and V-content in the metal versus lime addition.
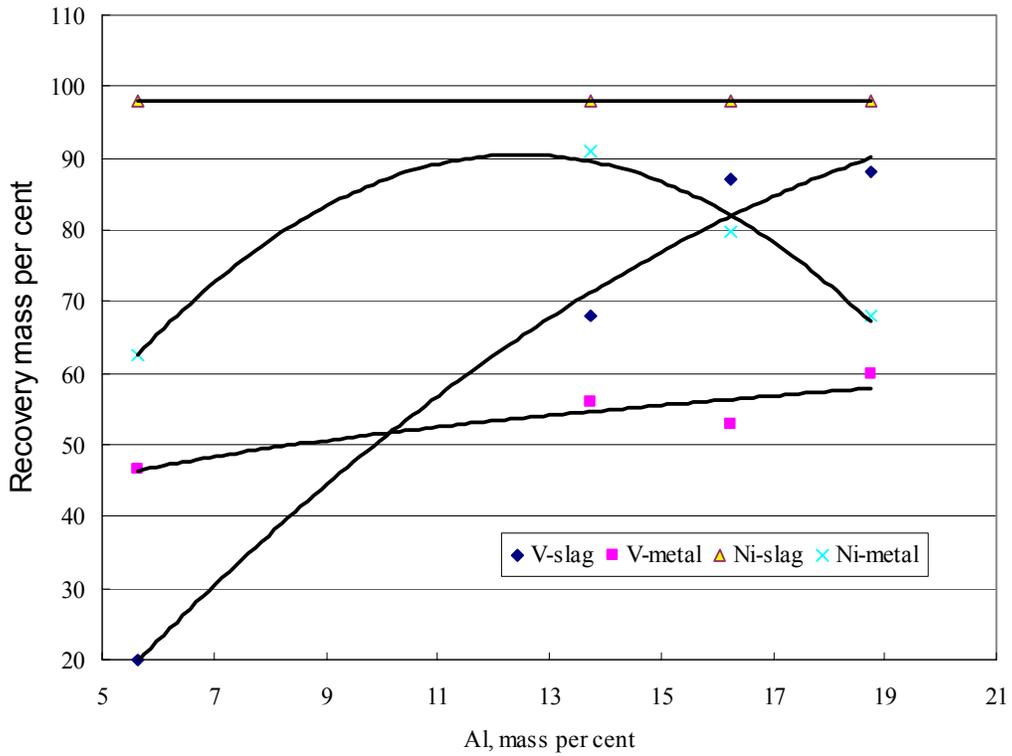

Figure 6. Vanadium and nickel recoveries versus aluminium addition based on masses and analyses of metals and slags produced.
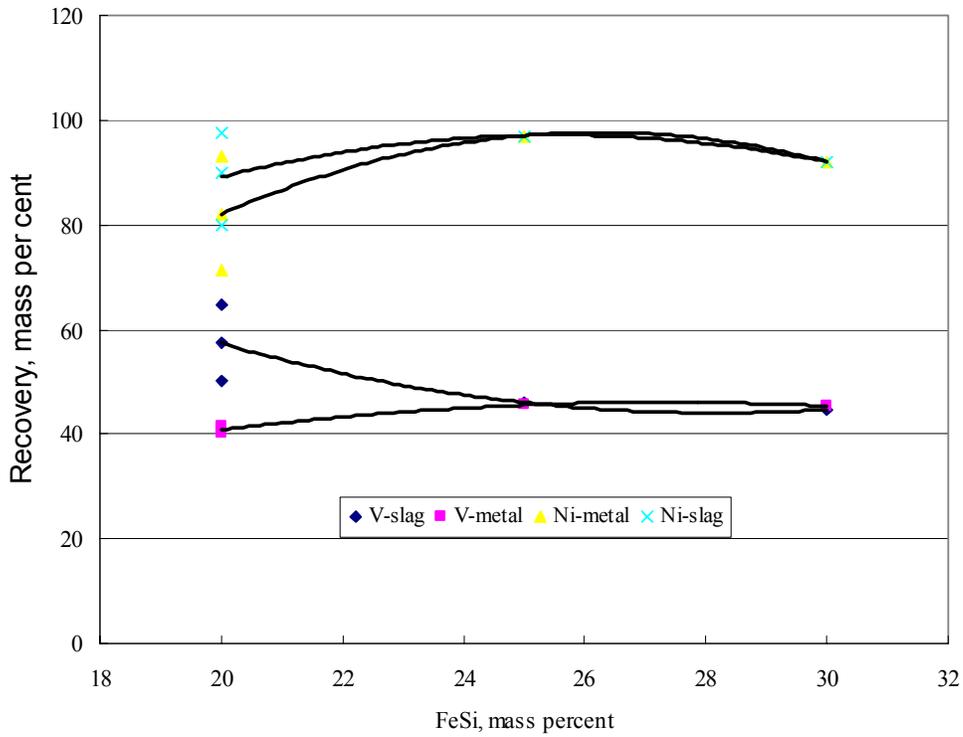
Figure 7. Vanadium and nickel recoveries versus FeSi addition based on masses and analyses of metals and slags produced.
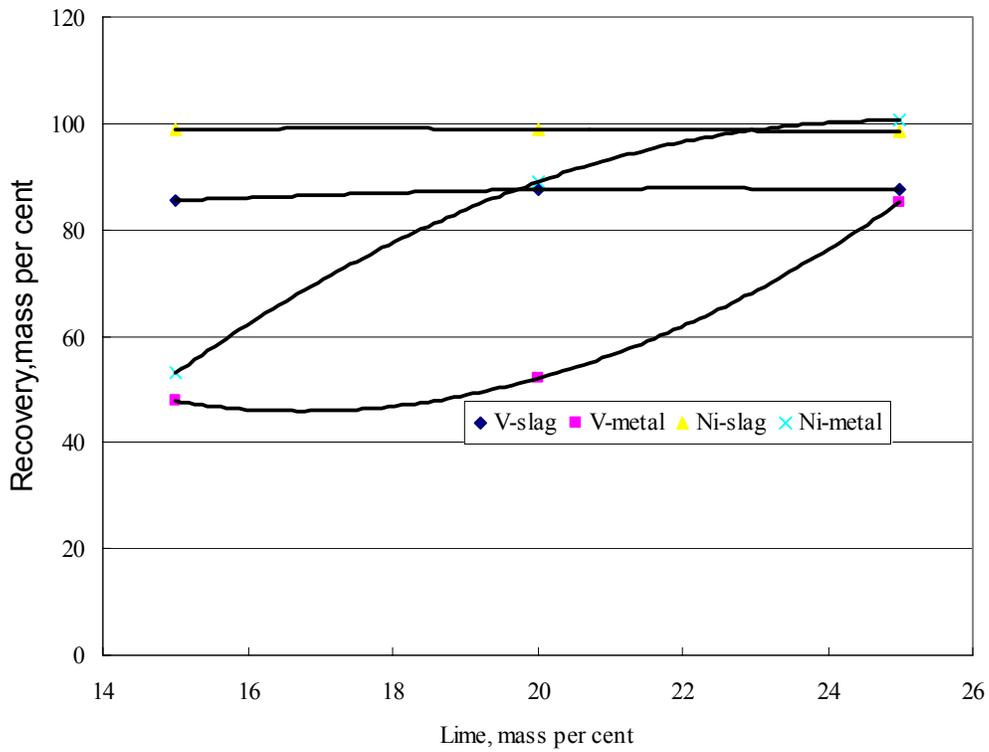

Figure 8. Vanadium and nickel recoveries versus lime addition based on masses and analyses of metals and slags produced.