

Behavior of Alloying Elements during Electro-Slag Remelting of Ultrahigh Strength Steel

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The paper deals with the behavior of principal constituents of an ultrahigh strength steels during ESR. The multiple analyses show no significant variation in the concentrations of silicon, manganese, vanadium, molybdenum, sulphur and phosphorus while concentrations of carbon, chromium, aluminium and titanium in the ESR ingot are found to be within the range of ± 0.01 , ± 0.12 , ± 0.008 and $\pm 0.01\%$ respectively. Silicon and manganese show a loss of 0-55 and 2-13% respectively whereas the degree of desulphurization is in the range of 0 (in Ar shield) to 52 (in air). The lower carbon and chromium content of the electrode than the aimed value can be corrected by adding ferrochromium in-situ during ESR. The recovery of carbon from FeCr was nearly 100% in most of the melts while the recovery of chromium varied over a fairly wide range. The distributions of carbon and chromium are also uniform along the height and width of ESR ingots. The recovery of the inoculant titanium was relatively low at the bottom of the ingot and it increases with height and reached a constant value above 100-150 mm height. For the same addition of titanium, its recovery decreases during ESR with argon shielding.

Keywords: ELECTROSLAG REMELTING, ULTRAHIGH STRENGTH STEEL, VARIATION IN ALLOY CONCENTRATIONS, RECOVERY, RESIDUAL INOCULANT.

Introduction

Electroslag remelting is used for production of steel for critical applications. ESR produces ingots with certain desirable characteristics almost independently of the metal melted like good ingot surface quality, a high yield, free from pipe, homogeneity of composition and improved cleanliness. ESR is primarily concerned with the remelting of ingots having a precise composition. Electro slag remelting (ESR) is used in the production of high-quality special steel, premium grade super alloys and titanium alloys for aerospace and other critical applications [1-10]. The ESR process has also been used to clad or join metals and to produce metal / metal or metal / ceramics composites [11].

The ingot to be produced by ESR is essentially of the composition of the electrode minus undesirable elements and conspicuous inclusions. Therefore, control of the chemistry in the ESR ingots is important to meet the stringent composition for achieving desirable properties – particularly for sensitive alloys where presence of essential elements is of small concentration often

less than a percent. For instance, in maraging steel 300 containing 0.8% Ti a loss of 0.1% will cause the yield strength to drop by up to 100 MPa [12].

Very few studies have been carried out to study the behaviour of alloying elements during ESR of Ultra High Strength Steel. Xiang et al studied the compositional homogeneity in an ESR ingot of 4.5 m height and 2.8 m diameter, produced in a 200 ton ESR facility. They reported that chemistry could be controlled within the range of $\pm 0.01\%$ C, $\pm 0.015\%$ Si and Mn, $\pm 0.02\%$ Cr and Mo, $\pm 0.001\%$ P, < 20 ppm S and < 30 ppm O [13].

The steel AFNOR 15CDV6 is a high strength bainitic steel (0.15%C) containing low concentrations of chromium (1.50%), molybdenum (0.8-1.00%) and vanadium (0.3%) as alloying elements. Because of its good strength-ductility combination and ease of fabrication, the material has been extensively used in rocket-motor hardware in the Indian space programme. By increasing the carbon and chromium content of the steel to 0.30 and 4% respectively and subjecting to inoculation with titanium during ESR, the 0.2%PS and ductility of the steel in as-cast condition and

after heat treatment has been increased to 1570-1610 MPa and 14% respectively[14].

The paper reports the behavior of alloying elements during ESR of AFNOR and modified 15CDV6 steel with particular reference to the effect of atmosphere on desulphurization, compositional adjustment and inoculation in-situ during ESR.

Methodology

In the present work, electrodes of standard (0.15-0.18C, 1.50Cr) and modified 15CDV6 steels with 0.27 – 0.31% carbon and 1.50 – 4.40% chromium were produced by melting mild steel scrap in a magnesia lined medium frequency (2650 Hz) air induction furnace of 20 kg capacity with additions of different ferro-alloys to adjust the composition to the required level. Finally, the steel was deoxidized in the furnace with Fe-Si (0.2% silicon addition) and in the ladle by the addition of 0.15 % aluminum and then cast into 40 mm diameter 600 mm long bars in vertical cast iron moulds. These bars were electro slag remelted using prefused slag of composition 70CaF₂–30Al₂O₃ (mass %) in a water-cooled four-piece split steel mould of 80 x 70 mm cross-section using DC electrode positive power. The current, voltage and average power input during ESR were maintained at 1300 A, 32 ± 2 V and 41 kW respectively in all the experiments.

The solidified slag was crushed just before ESR and screened to below 20 mesh size powder and preheated at 700–800°C for 5-6 hours in a muffle furnace to remove any moisture present. The amount of slag used in each experiment was 1 kg which gave a slag bath depth of about 50 mm. During ESR, the current, voltage and average power input were maintained at 1300 A, 32 ± 2V and 41kW respectively in all the experiments. In all the ESR heats, the slag was deoxidized by continuous addition of aluminum (0.10-0.20% of the electrode weight) in the form of 3 mm diameter wire tied along the length of the electrode being refined. The cooling water flow rates in the mould jacket and the base plate were maintained at 40 and 25 liters per minute respectively. The temperature of the outlet water from the mould was 35 + 1.5°C. Titanium in the form of ferro-alloy powders (FeTi: Ti 35%, Al 5%;) of below 60 mesh size, obtained by crushing and grinding lumps, were used as inoculants in the investigation. Thin walled mild steel tube (OD = 6mm, ID = 4 mm) containing the requisite amount of the inoculant powder was sealed at both the ends and tack welded along the

length of the electrode remelted.

In some of the experiments, the carbon and chromium contents in the electrodes were slightly lower than the aimed levels in the ESR ingots. This was corrected by the addition of requisite quantities of high carbon ferro-chrome powder (composition: Cr 60- 5%, C 6– 8%, Si 2%; size: – 60 mesh) along with the inoculant through the mild steel tube.

The cross-sections and heights of the ingots produced were in the range of 75 x 65 to 78 x 68 mm and 180 to 300 mm respectively. The weight of the ESR ingots was in the range of 7-12 kg. The ESR ingots were homogenized for six to seven hours in a muffle furnace. During homogenization the temperature was measured at various points near the ingot. In all the experiments, the temperature near the middle of the ingot was measured to be 975°C whereas temperatures near the ends varied between 950° and 960°C. After homogenization, 10 mm from the top and 20 mm from the bottom of the ingots were cut and discarded. Only the remaining part of the ingot was used for the various studies. The scheme of sampling for chemical analysis is shown in **Figure 1**.

Samples taken from top and bottom locations of the ingots were analyzed for various elements. Analysis for residual inoculant's content was carried out on samples at 90 mm height from the bottom of the ingot. The methods used for analysis of the various elements in the steels are listed below: The alloying elements were analyzed by vacuum spectrograph. Titanium was also analyzed both by Atomic Emission Spectrophotometer and Inductively Coupled Plasma-Atomic Absorption Spectrophotometer

Results and Discussion

Four different compositions of steel i.e. standard 15CDV6 and modified 15CDV6 steels with higher carbon and higher chromium contents were produced by ESR with and without inoculation. The ESR ingots were free from any shrinkage, pipe or other solidification defects. The ingots had a smooth surface covered with a uniform slag skin of 1-2 mm thickness. The chemical compositions of the electrodes used for ESR and of the ESR ingots produced are given in Table1. Values reported in the tables are the average of four values, that is, two from the top and two from the bottom of the ingot. In all the cases these four values were close to each other.

Comparison of the compositions of the ESR

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ingots and those of the electrodes (**Table 1**) showed that there was practically no change in the concentrations of carbon, molybdenum, vanadium and phosphorus during ESR.

Table 1. Nominal chemical composition of electrodes and ESR ingots

	C	Si	Mn	P	S	Cr	V	Mo	Al
El	0.16	0.05	0.86	0.019	0.022	1.48	0.27	1.08	0.023
In1	0.16	0.05	0.80	0.017	0.015	1.48	0.27	1.03	0.022
El	0.16	0.06	0.87	0.015	0.022	1.52	0.30	1.02	0.010
In2	0.15	0.05	0.83	0.017	0.012	1.50	0.29	1.03	0.065
In3	0.15	0.05	0.80	0.016	0.012	1.53	0.29	1.05	0.039
El	0.29	0.11	0.83	0.022	0.016	1.50	0.27	0.95	0.011
In4	0.29	0.04	0.80	0.024	0.010	1.50	0.27	0.96	0.023
In5	0.30	0.05	0.81	0.023	0.010	1.50	0.27	0.94	0.035
El	0.27	0.07	0.77	0.015	0.014	1.68	0.21	0.96	NA
In6*	0.29	0.05	0.71	0.019	0.010	1.65	0.21	0.96	0.084
El	0.28	0.09	0.78	0.019	0.019	1.76	0.28	0.82	0.016
In7*	0.30	0.06	0.74	0.021	0.012	1.90	0.26	0.82	0.093
In8*	0.29	0.08	0.72	0.021	0.019	1.74	0.26	0.82	0.098
El	0.25	0.18	0.70	0.013	0.021	3.44	0.21	0.98	0.027
In9+	0.31	0.16	0.67	0.016	0.015	4.12	0.21	0.94	0.098
El	0.26	0.15	0.84	0.019	0.025	4.39	0.32	0.81	0.051
In10#	0.28	0.11	0.71	0.021	0.014	4.40	0.29	0.79	0.042
El	0.26	0.19	0.80	0.022	0.025	4.05	0.27	0.70	0.051
In11#	0.27	0.15	0.75	0.034	0.012	4.08	0.28	0.68	0.055

* Note: El: electrode; In: ESR ingot produced. High carbon FeCr amounting to * 0.29 %, + 0.72% and # 0.41% of the weight of the electrode was added during ESR.

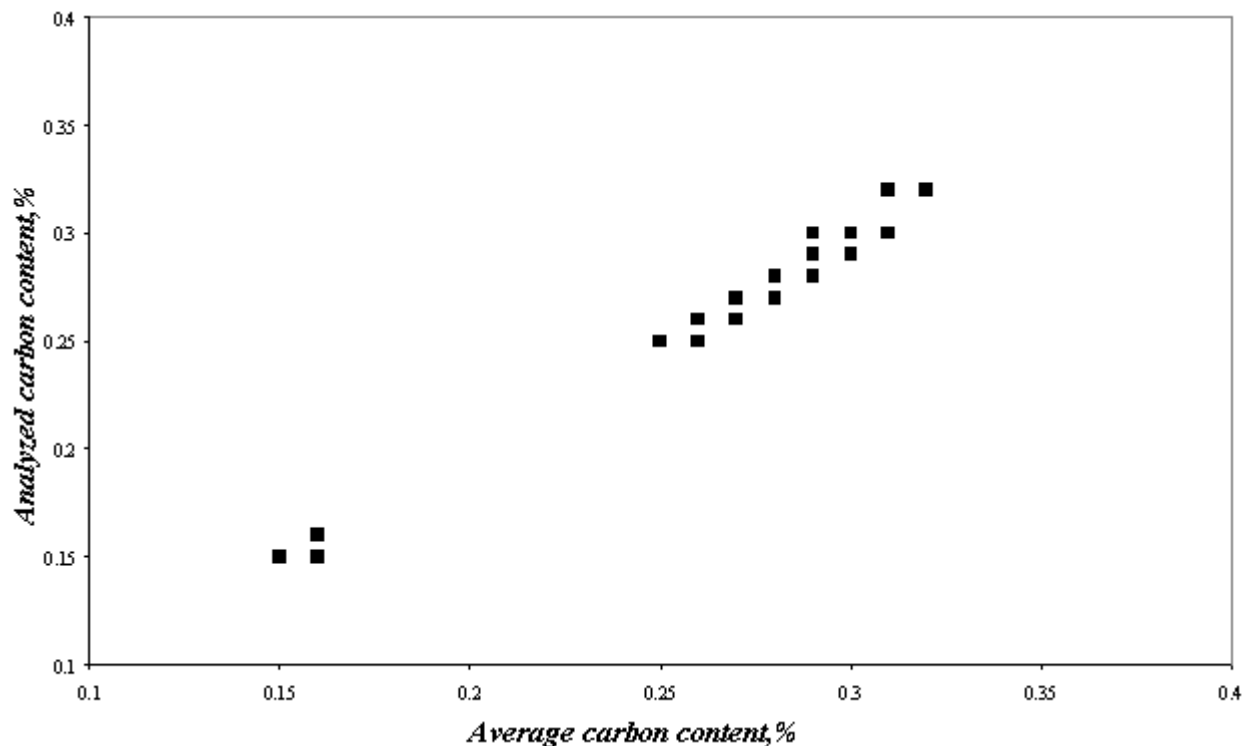


Figure 2. The variation in the analysis of carbon at various concentrations

Duplicate/multiple chemical analyses were carried out on several samples from electrodes and ESR ingots in order to check the reproducibility of

chemical analyses. The multiple analyses show no significant variation in the concentrations of silicon, manganese, sulphur and phosphorus.

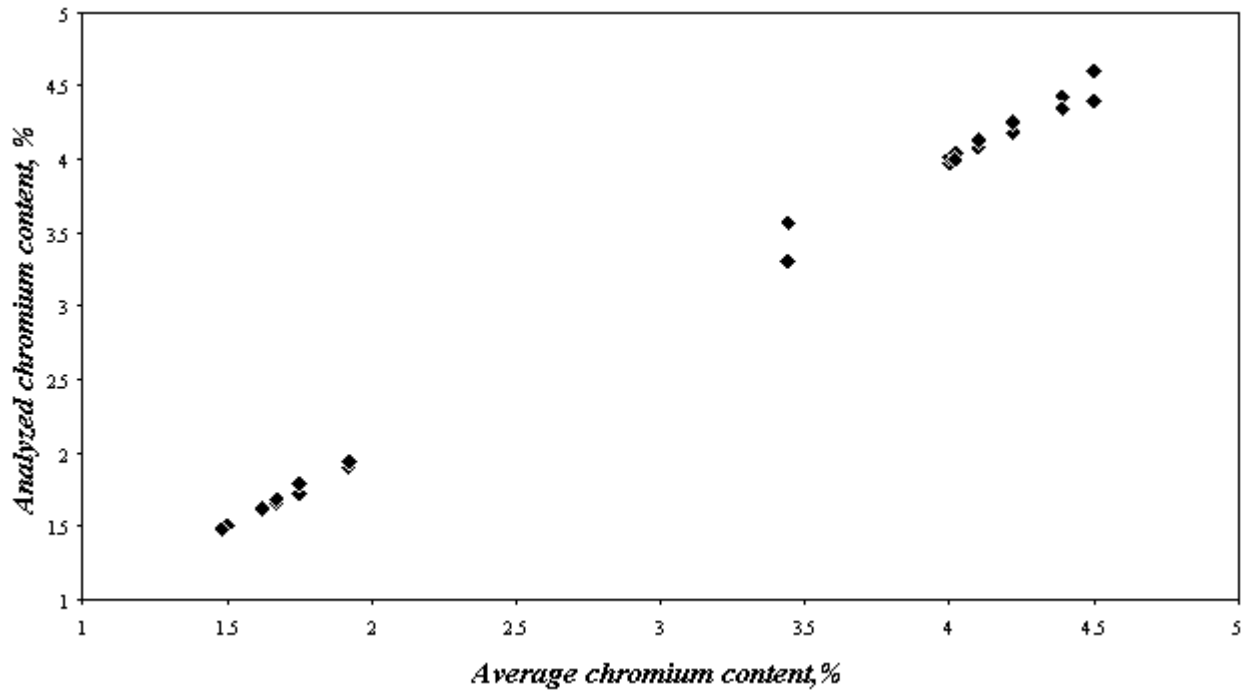


Figure 3. The variation in the analysis of chromium at various concentrations

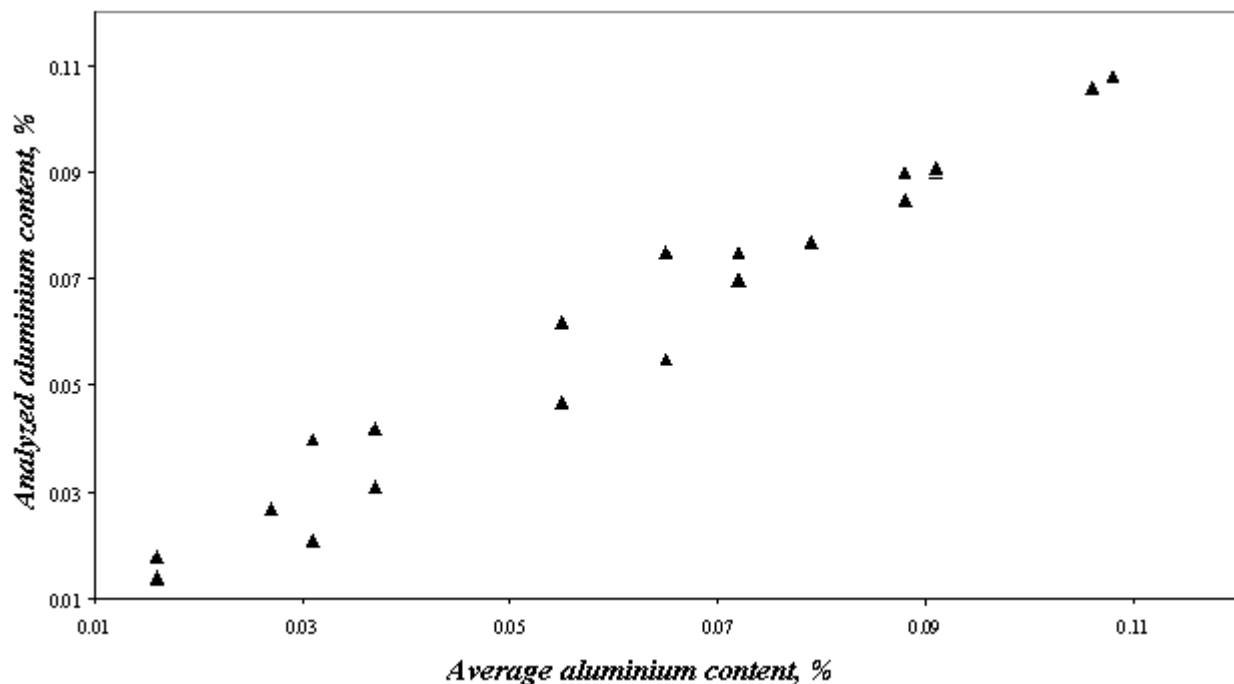


Figure 4. The variation in the analysis of aluminium at various concentrations

However, there was some variation in the values of carbon, chromium, aluminum and titanium. For carbon, aluminum and titanium the variations occurred within a band of constant width irrespective of average content. However, the width of the band showing the variation in chromium increased slightly with increasing chromium content. The variations observed for the carbon, chromium, aluminum and titanium are

shown in **Figures 2 to 5** and given in **Table 2**.

Table 3 shows the changes in Si and Mn content of the steels after ESR. The decrease in the manganese content during ESR was in the range of 2-13% and that of silicon varied over a wider range of 0-55% as indicated in the Table. The standard free energy for the formation of SiO_2 (-562 kJ at 1973K) is higher than that of Al_2O_3 (-699 kJ at 1973K) involving pure oxide with unit activity.

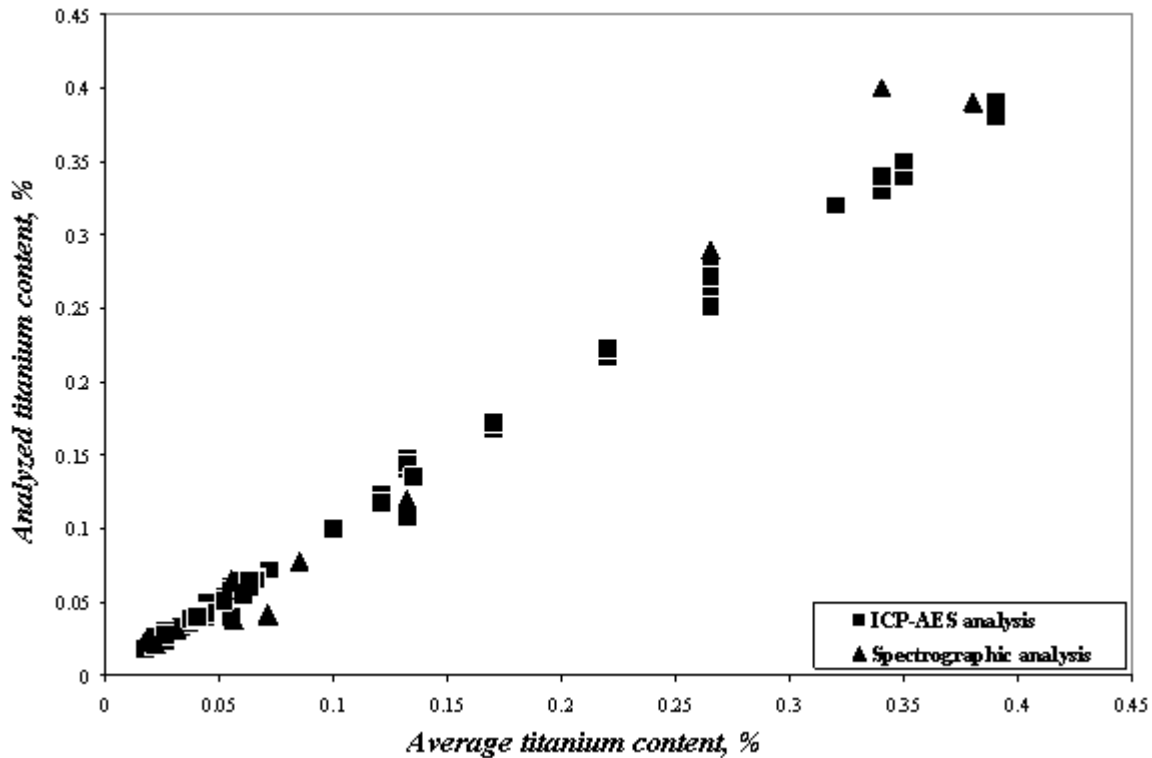


Figure 4. The variation in the analysis of titanium at various concentrations

Table 2. The range of scatter observed in the analyzed concentration of elements

Element	Range of variation (%)
Carbon	0.02
Chromium	0.16 (at 1.5 – 2% Cr) 0.24 (at 3.5 – 4.5%Cr)
Aluminium	0.016
Titanium	0.02

And it is expected that Si in the electrode will be oxidized primarily by the FeO formed in the slag due to dissolution of oxygen from the atmosphere. Chemical reactions during ESR are governed basically by the interaction between slag and meal. Since the ESR slag contains CaF₂ (70%)-Al₂O₃ (30%) by weight, the activity of (Al₂O₃) increases and that of (SiO₂) decreases due to formation of CaO as high as 5% by pyrohydrolysis of CaF₂ [15]. Therefore, the more loss of silicon can be attributed to the reaction $3 [Si] + 2(Al_2O_3) = 4 [Al] + 3(SiO_2)$.

In electrodes for In 6 to In 11, the carbon content was lower than the aimed value in the ingot. This was attempted to be corrected by the addition of high carbon ferrochrome continuously

during ESR. The recoveries of carbon and chromium from the high carbon ferrochrome added during ESR are shown in Tables 4 and 5 respectively. The recovery of carbon was nearly 100% in most of the melts while the recovery of chromium varied over a fairly wide range.

Two ESR ingots (In 9 and In11), in each of which high carbon ferrochrome was added during ESR in order to increase the carbon and chromium content of the metal, were analyzed at a number of locations to study the uniformity of composition in the ingot. The distribution of carbon in the ESR ingots 9 and 11 is shown in Figure 6. It can be seen from the figure that addition of high carbon ferrochrome during ESR was effective in increasing the carbon content of the electrode to the desired value in the ingot and distribution of carbon was also uniform along the height of the ESR ingots. There was a slight variation in the chromium content along the height and width of the ingot (Figure 7). Chromium content varied from 4.00 to 4.40% in ESR ingot 9 and from 3.95 to 4.23 in ESR ingot 11.

There was also a decrease in the concentration of sulphur in the metal during ESR as expected. The degree of desulphurization during ESR was to the extent of 30–50% (Table 6). However, in one melt (ESR 8) in which argon shielding was employed inside the ESR mould, there was no desulphurization. This may be attributed to the lack

Table 3. Change in the silicon and manganese contents of the metal during ESR

ESR melt No.	% in electrode		% in ingot		Loss (%)	
	Si	Mn	Si	Mn	Si	Mn
In 1	0.05	0.86	0.05	0.80	0	07
In 2	0.06	0.87	0.06	0.84	0	03
In 3	0.06	0.87	0.05	0.80	17	08
In 4	0.11	0.83	0.05	0.81	55	02
In 5	0.11	0.83	0.06	0.81	45	02
In 6	0.07	0.77	0.05	0.71	29	08
In 7	0.09	0.78	0.06	0.74	33	05
In 8	0.09	0.78	0.08	0.72	11	08
In 9	0.18	0.70	0.10	0.68	44	03
In 10	0.15	0.84	0.11	0.73	27	13
In 11	0.19	0.80	0.15	0.75	21	06

Table 4. Recovery of carbon from high carbon ferrochrome added during ESR

% C in the electrode	Aimed % C in the ingot (ESR No)	Amount of HCFeCr# added (% of electrode mass)	% C added in the form of HCFeCr	% C in the ESR ingot	Recovery of Carbon (%)
0.27	0.29 (In6)	0.29	0.02	0.29	100
0.28	0.30 (In7)	0.29	0.02	0.30	100
0.28	0.30 (In8)	0.29	0.02	0.30	100
0.25	0.30 (In9)	0.72	0.05	0.32	140
0.26	0.29 (In10)	0.41	0.03	0.29	100
0.26	0.29 (In11)	0.41	0.03	0.27	33

#HCFeCr – High carbon ferrochrome (65%Cr, 7 % C).

of oxidation of the sulphur content of the slag by the atmosphere.

In all the ESR melts, the slag was deoxidized by continuous addition of aluminum in the form of a wire attached to the electrode. In several melts there was a pick up of aluminum by the metal during ESR (**Table 7**). In general, the pick up was larger with increase in aluminum addition. The

amount of aluminum added was between 0.1-0.2% of the weight of the metal. The aluminum content in the electrode was in the range of 0.01-0.05%. When aluminum amounting to 0.10% of the weight of the ingot was added to the slag, the aluminum content in the metal increased from 0.01% to a value of 0.02-0.04%. On increasing the aluminum addition to 0.2% of the weight of the ESR ingot,

Table 5. Recovery of chromium from high carbon ferrochrome added during ESR

% Cr in the electrode	Amount of HCFeCr# added (% of electrode mass)	% Cr added in the form of HCFeCr	Range of % Cr in the ESR ingot	Range of chromium recovery (%)
1.68	0.29 (In-6)	0.19	1.64 – 1.65	0
1.76	0.29 (In-7)	0.19	1.88 – 1.92	63 – 84
1.76	0.29 (In-8)	0.19	1.67 – 1.82	0 – 32
3.44	0.72 (In-9)	0.47	4.00 – 4.40	119 - 204
4.39	0.41 (In-10)	0.27	4.21 – 4.50	0 – 41
4.05	0.41 (In-11)	0.27	3.95 – 4.20	0 – 56

HCFeCr – High carbon ferrochrome (65 % Cr, 7 % C)

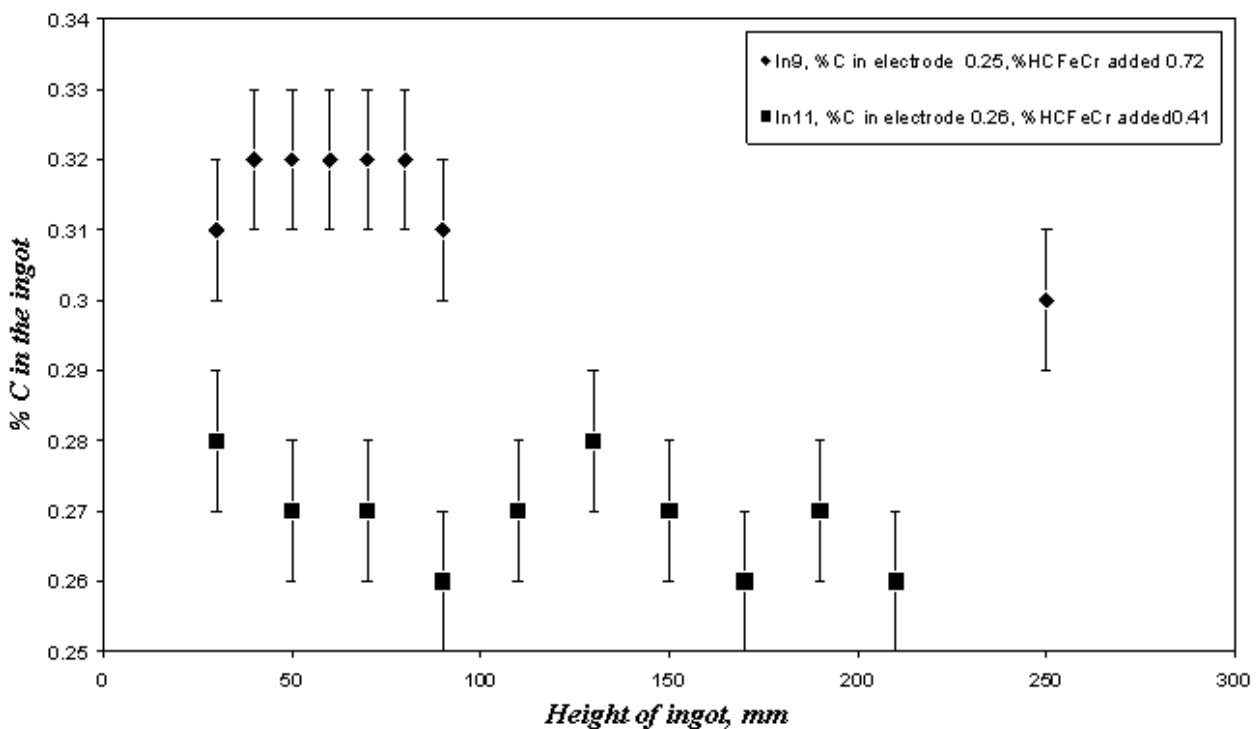


Figure 6. Distribution of carbon content along the height of the ESR ingots (9 & 11)

the aluminum content in the metal increased from 0.02-0.05% in the electrode to about 0.09% in the ESR ingot. The mechanism of aluminum pick up by the ESR metal is not apparent. The aluminum added is expected to reduce the FeO content of the slag. Any excess aluminum remaining after reducing the slag will float on the surface of the slag since the density of the liquid aluminum (density at m.p. 2375 Kg^m⁻³) is lower than that of the ESR slag (density at 1650^oC 2800 Kg^m⁻³). This

layer of excess liquid aluminum will be oxidized to Al₂O₃ by the atmosphere. Hence it is difficult to conceive that any of the aluminum added for deoxidation of the slag will be able to directly enter the metal. However, reactive elements like silicon and titanium may reduce alumina in the slag at the slag metal interface leading to the increase in the aluminum content of the metal. These reactions may become feasible through the formation of TiO as an intermediate product since

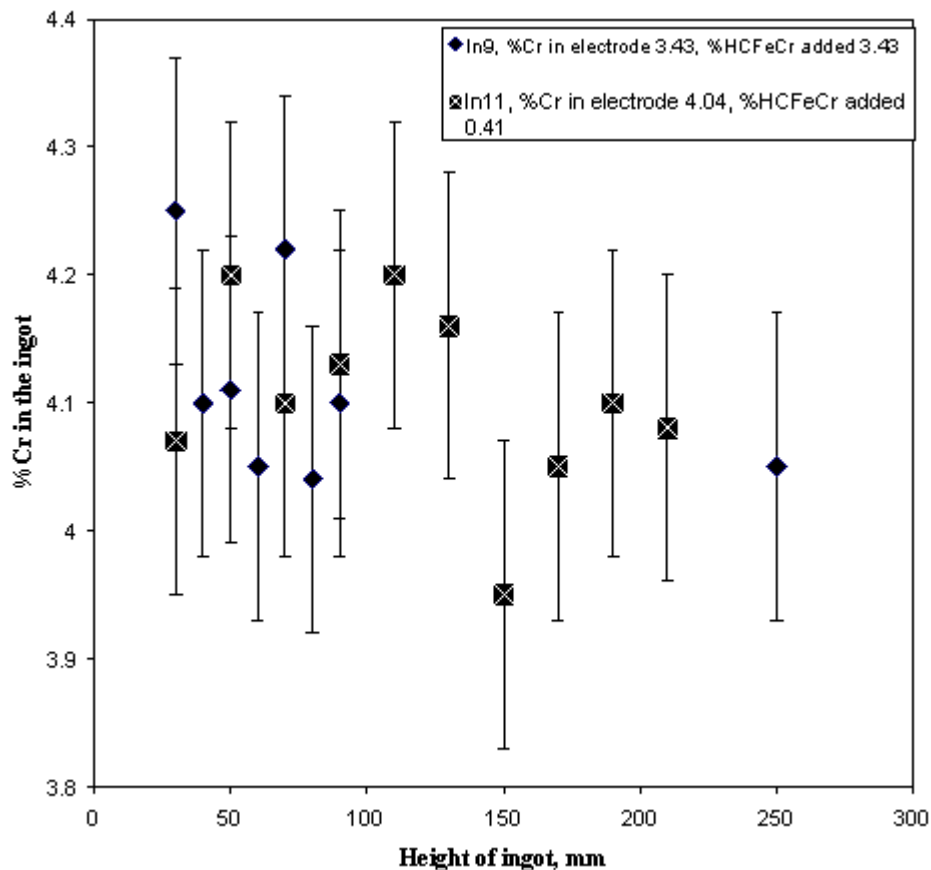


Figure 7. Distribution of chromium content along the height of ESR ingots (In 9 & 11)

Table 6. Sulphur content of steels before and after ESR

Material	S (wt%)	Degree of desulphurization
Electrode	0.022	--
In 1	0.015	32
In 2	0.012	45
In 3	0.012	45
Electrode	0.016	--
In 4	0.010	38
In 5	0.010	38
Electrode	0.014	--
In 6	0.010	29
Electrode	0.019	--
In 7	0.012	37
In 8	0.019	0
Electrode	0.021	--
In 9	0.015	29
Electrode	0.025	--
In 10	0.014	44
Electrode	0.025	--
In 11	0.012	52

the standard free energy of formation of TiO (-677 kJ at 1973K) is comparable to that of Al₂O₃ (-699 kJ at 1973K) and favored by very low activity of TiO₂ and high activity of Al₂O₃ in the ESR slag

[16]. It has also been suggested that formation of TiO as an intermediate product may facilitate the slag-metal reactions, since the free energy of formation of TiO may be comparable to the free energy of formation of SiO₂ [17].

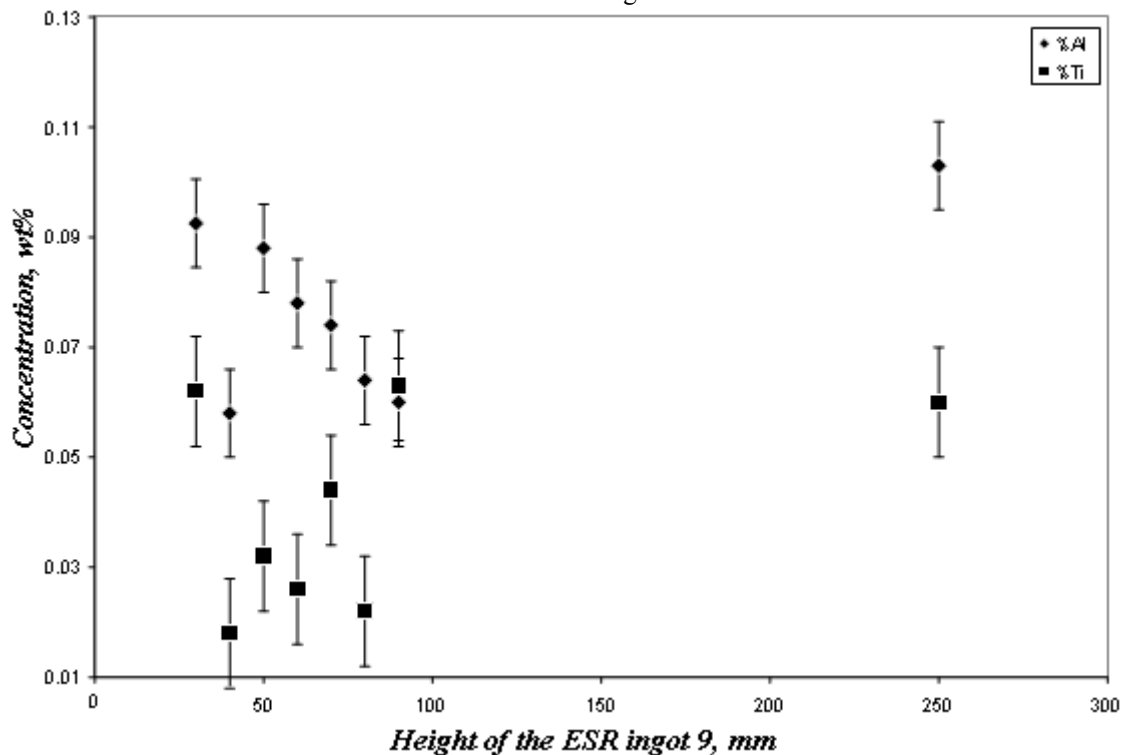
Figures 8 and 9 show the distribution of aluminum along the height of the remelted ESR ingots (In 9 and In 11). It can be seen that the distribution of aluminum along the ingot height is not very uniform. From these data (Figures 8 and 9), there seems to be a clear trend towards a lower Ti content at the bottom of the ingot, which increased and stabilized after a height of about 100 mm from the bottom of the ingots. This may be caused by the absence of a stable deoxidized slag bath during the start up and initial stages of the ESR melt resulting in loss of titanium due to oxidation. The upper part of the ingot represents stable melting condition with very little oxidation.

One interesting observation is the effect of argon shielding on residual titanium and aluminum content in the case of In 8. The residual titanium content (0.026-0.17%) and percent of recovery of titanium (7-43) in ESR Ingot 8 with Ar shielding (In 8) are much lower than that of in ESR Ingot 6 (In 6) i.e. 0.13-0.278% and 33-70 respectively with the same addition of titanium i.e. 0.4wt% in open

Table 7. Pick up of aluminium by the metal during ESR

ESR Melt No.	% Al in electrode	% Al added during ESR	% Al in ingot		Pick up of Al, %	
			Top	Bottom	Top	Bottom
In 1	0.023	0.15	0.021	0.023	0	0
In 2	0.010	0.14	0.082	0.046	51	26
In 3	0.010	0.10	0.036	0.042	26	32
In 4	0.011	0.10	0.020	0.025	09	14
In 5	0.011	0.10	0.034	0.036	23	25
In 6	--	0.20	0.077	0.091	--	--
In 7	0.016	0.21	0.079	0.108	30	44
In 8	0.016	0.21	0.088	0.108	34	44
In 9	0.027	0.18	0.103	0.092	42	36
In 10	0.051	0.20	0.031	0.053	0	0
In 11	0.051	0.20	0.058	0.052	4	0

Figure 8. Distribution of Al & Ti along the height of ESR ingot 9, deoxidised with 0.1wt% Al and inoculated with 0.2wt%Ti during ESR



atmosphere. But the residual aluminum content in the ESR ingot 6 i.e. 0.077-0.091% is higher than that of in ESR ingot 8 i.e. 0.089-0.1085% with the

same addition of Al (0.20-0.21%). This may be attributed to the fact that at low oxygen potential of the slag (FeO) caused by Argon shielding Ti is

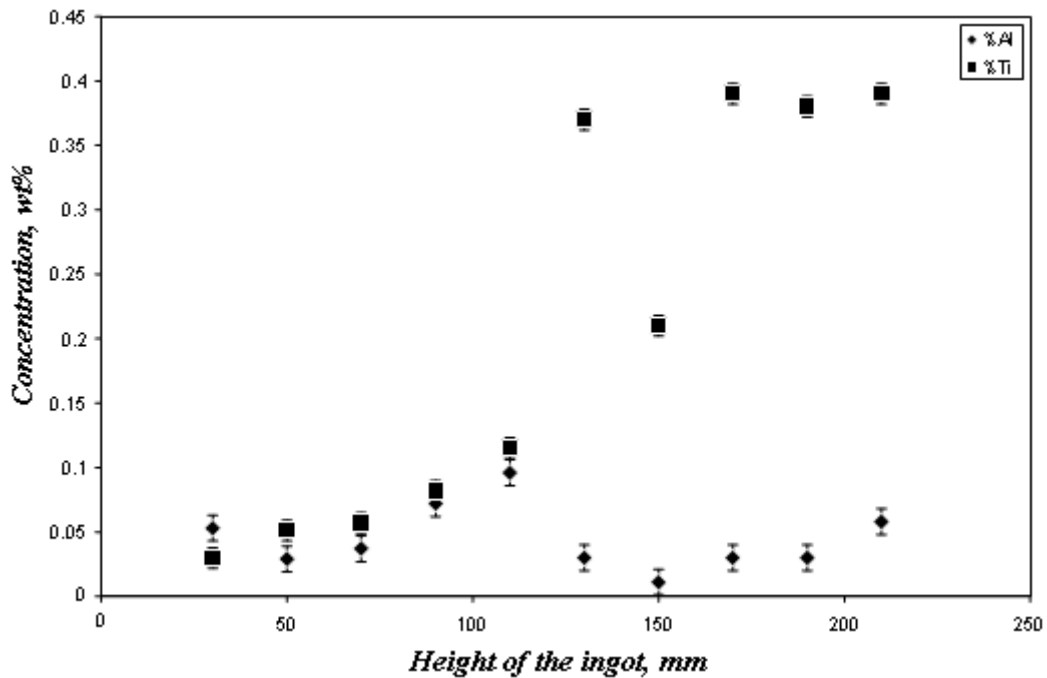


Figure 9. Distribution of Al & Ti along the height of ESR ingot 11, deoxidised with 0.1wt% Al and inoculated with 0.4wt%Ti during ESR

more prone to oxidation than Al which is further facilitated by the low concentration of TiO_2 in the slag as discussed in the preceding paragraph.

Conclusions

1. During ESR, the carbon, chromium, aluminum and titanium contents in the ingot can be controlled within ± 0.01 , ± 0.12 , ± 0.008 and $\pm 0.01\%$ respectively.

2. During ESR, it is possible to add high carbon/low carbon ferrochrome through mild steel tube attached to the electrode to increase the carbon and/or chromium content of the metal. The recovery of carbon can be very high while the recovery of chromium is variable.

3. The recoveries of carbon and chromium are uniform along the height and width of ESR ingots.

4. Addition of ferro-titanium/ferro-niobium through mild steel tube attached to the electrode during ESR is an effective method of inoculating steel with titanium and niobium. The recovery of inoculant increases with increasing addition of inoculant. For example, when the inoculant addition was 0.15-0.20% of the weight of the metal its recovery was 18-70% which increased to 40-100% when the addition was 0.4%.

5. The recovery of the inoculant titanium was relatively low at the bottom of the ingot and it increases with height and reached a constant value above 100-150 mm height.

6. For the same addition of titanium, its

recovery decreases during ESR with argon shielding.

7. The recovery and hence, the distribution of aluminum along ESR ingots is not very uniform.

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