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Fusion Behaviour of Synthetic Aluminothermic Ferro-chrome Slags

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Fusion behaviour of synthetic slags resembling those produced during Alumino-thermic reduction of chromite ore for production of Low-carbon ferro-chrome has been thoroughly discussed.

The effect of fluxes like CaO, MgO and CaF_2 on the measured values of the characteristic temperatures, which represent the fusion behaviour of a slag, has been experimentally determined and critically discussed.

All the three fluxing constituents are found to decrease the characteristic temperatures. CaF_2 is found to decrease the gap between the liquidus temperature and the temperature characterising the flow of the slag.

KEY WORDS: fusion-behaviour; aluminothermic-reduction; characteristic temperatures; slag–metal separation; hemispherical temperature; liquid mobility; flow units; long-slag, short-slag.

1. Introduction

The slag generated from a pyrometallurgical process should have a low fusion-temperature so that at the furnace operating temperature a high available super-heat will increase the kinetics of chemical reactions and will ensure better slag–metal separation. It is in this context that many investigations have been carried out in the past to study the effect of compositional variations on liquidus temperature.[1–5] However, behaviour of a single constituent in a multi-component slag is different in different circumstances due to its interaction with other constituents. Hence it is essential to study the liquidus temperature of each particular slag resulting from a specific process.

The liquidus temperature has been reported by various nomenclature by various investigators. Osborn[6] and Snow[7] reported liquidus temperature as the temperature at which the first crystal forms during cooling down the melt whereas Ohno *et al.*[8] reported it as the temperature at which the last crystal disappears during heating. The liquidus temperature or fusion point in German Industrial Standards 51730 has been defined as the hemispherical temperature, *i.e.*, the temperature at which a small mass of the slag assumes the shape of a hemisphere on heating. The heating microscope method is adopted to measure this hemispherical temperature.

In industrial melts, *i.e.*, when a pyrometallurgical process is carried out in a furnace, it is the fusion behaviour of the non-metallic melts (*i.e.*, the mixture of oxides which would combine to make the slag) which is more important than the exact fusion temperature. Enough data are not available pertaining to the fusion behaviour of slags generated in various smelting processes though its relevance *vis-à-vis* the process can not be denied. The fusion behaviour is described in terms of four characteristic temperatures. These are; the initial deformation temp. (IDT) symbolising the surface stickiness; the softening temp. (ST), symbolising the plastic distortion; the hemispherical temperature (HT), which is also the liquidus temperature, symbolising sluggish flow; and the flow temperature (FT), symbolising liquid mobility. The fusion behaviour of the slag is very relevant in smelting and refining processes. The IDT, indicating the surface stickiness of the material is important for movement of the material in the solid state. Similarly the ST, which indicates the start of plastic distortion along with the HT which represents the fusion or liquidus temperature of the material, plays a significant role in the aerodynamics and heat and mass transfer. In a Blast furnace, for example, a high softening and a high fusion temperature of iron bearing materials would lower the cohesive (softening-melting) zone in the furnace thus decreasing the distance travelled by the molten metal droplets in the furnace there by decreasing the silicon pick-up.[9–11]

Both, the HT as defined in the previous paragraph, and FT which symbolises the start of liquid mobility are very important in any smelting refining process starting with solid/solid and liquid materials and producing the final product in the liquid state. Lower HT and FT indicate low energy expenditure. The aluminothermic reduction process for production of extra low carbon ferro-alloys or pure metals is a very fast process and requires high liquid mobility of slag and metal for better slag–metal separation. Hence the liquidus temperature (HT) and the flow temperature (FT) both should be as low as possible for higher yield of the metal/alloy.

The characteristic behaviour of a slag under any operating conditions is a function of its composition and the various constituents present in it of which the fluxing constituents are the most important ones. It is in this context that an attempt has been made to study the effect of various fluxing agents on the fusion behaviour of the concerned slags with particular emphasis on its effect on the HT and the FT.

2. Experimental

2.1. Composition of Slags and Method of Synthetic Slag Preparation

In the first stage 37 nos. of slags for which the viscosity data has already been published[12,13] were selected also for a systematic study of the fusion behaviour. The compositional details of these slags are presented in **Tables 1** through **6**. These slags resemble the composition of slags encountered in the industry.

The first 2 groups of slags did not contain any CaF_2. While the Al_2O_3/CaO ratio for the first group of 8 nos. of slags was varied from 1.4 to 2.6, for the second group of 5 nos. of slags the MgO content was varied from 4 to 12 wt% with the Al_2O_3/CaO ratio being fixed at 2.5.

The rest 24 nos. of slags were put under four groups with Al_2O_3/CaO ratio being 2.0, 2.5, 3.0 and 3.4 for the respective groups. In each of these groups, consisting of 6 nos. of slags, CaF_2 was varied from 0 to 12.0 wt% in steps of 2.0 wt%. In all these slags the other constituents, namely, SiO_2, FeO, Na_2O and Cr_2O_3 are adjusted with minor variations to make the total 100 wt%. Such minor variations are well within the range of composition of the slags generated in industry.

Table 1. Compositional details of slags of Gr. 1. (variation of Al_2O_3/CaO wt. ratio)

COMPOSITIONAL DETAILS									
Slag No.	**Al_2O_3**	**CaO**	**MgO**	**Na_2O**	**Cr_2O_3**	**SiO_2**	**FeO**	**CaF_2**	**Al_2O_3/CaO**
1	57.98	22.3	4.3	5.3	4.5	2.80	2.82	-	2.60
2	58.25	24.27	4.2	5.05	4.4	1.9	1.9	-	2.40
3	56.3	26.7	4.1	5.0	4.3	1.80	1.80	-	2.10
4	56.25	28.0	3.29	4.56	4.29	1.64	1.64	-	2.00
5	55.37	30.6	3.15	4.43	3.21	1.57	1.57	-	1.80
6	56.48	31.48	2.78	3.7	2.78	1.39	1.39	-	1.99
7	53.0	34.0	3.0	4.0	3.0	1.50	1.50	-	1.56
8	51.1	36.40	2.88	3.86	2.88	1.44	1.44	-	1.40

Table 2. Compositional details of slags of Gr. 2. (variation of MgO at $Al_2O_3/CaO=2.5$)

PERCENTAGE COMPOSITION								
Slag No.	**Al_2O_3**	**CaO**	**MgO**	**Na_2O**	**Cr_2O_3**	**SiO_2**	**FeO**	**CaF_2**
9	60.42	24.16	4.0	4.67	3.55	1.6	1.6	-
10	58.63	23.44	6.0	4.75	3.82	1.68	1.68	-
11	58.13	23.25	8.0	4.34	3.4	1.44	1.44	-
12	56.66	22.66	10.0	4.45	3.25	1.50	1.50	-
13	55.0	22.0	12.0	4.5	3.5	1.5	1.50	-

Table 3. Compositional details of slags of Gr. 3. (CaF_2 variation at $Al_2O_3/CaO=3.4$)

PERCENTAGE COMPOSITION								
Slag No.	**Al_2O_3**	**CaO**	**MgO**	**Na_2O**	**Cr_2O_3**	**SiO_2**	**FeO**	**CaF_2**
14	62.00	18.23	2.50	4.90	5.40	3.40	3.40	0
15	60.78	18.87	2.45	4.80	5.30	3.40	3.40	2
16	59.60	17.52	2.40	4.60	5.20	3.32	3.36	4
17	58.49	17.20	2.35	4.30	5.10	3.30	3.26	6
18	57.40	16.88	2.31	4.00	5.00	3.22	3.18	8
19	56.36	16.57	2.27	3.70	4.87	3.08	3.15	10

Table 4. Compositional details of slags of Gr. 4. (CaF_2 variation at $Al_2O_3/CaO=3.00$)

PERCENTAGE COMPOSITION								
Slag No.	**Al_2O_3**	**CaO**	**MgO**	**Na_2O**	**Cr_2O_3**	**SiO_2**	**FeO**	**CaF_2**
20	60.00	20.00	2.50	5.00	5.50	3.50	3.50	0
21	58.80	19.60	2.45	4.90	5.39	3.43	3.43	2
22	57.69	19.23	2.40	4.75	5.20	3.36	3.36	4
23	56.56	18.86	2.30	4.70	5.18	3.20	3.20	6
24	55.55	18.51	2.31	4.63	5.00	3.00	3.00	8
25	54.90	18.30	2.31	4.62	5.10	2.39	2.39	10

Table 5. Compositional details of slags of Gr. 5. (CaF_2 variation at $Al_2O_3/CaO=2.5$)

PERCENTAGE COMPOSITION								
Slag No.	**Al_2O_3**	**CaO**	**MgO**	**Na_2O**	**Cr_2O_3**	**SiO_2**	**FeO**	**CaF_2**
26	58.00	23.00	2.00	6.00	5.00	3.00	3.00	0
27	56.87	22.55	1.92	5.88	4.90	2.94	2.94	2
28	55.77	22.12	1.90	5.75	4.70	2.88	2.88	4
29	54.71	21.70	1.88	5.55	4.60	2.83	2.83	6
30	54.53	21.30	1.85	5.50	4.63	2.10	2.10	8
31	52.75	21.10	1.90	4.95	4.90	2.45	1.95	10

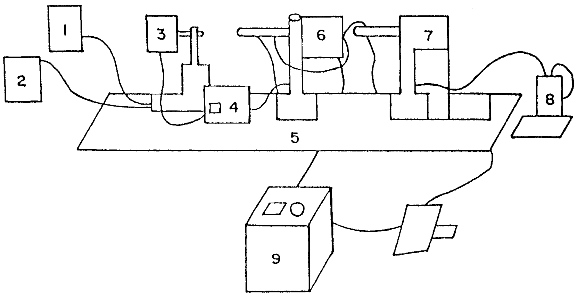
Table 6. Compositional details of slags of Gr. 6. (CaF_2 variation at $Al_2O_3/CaO=2.00$)

PERCENTAGE COMPOSITION								
Slag No.	**Al_2O_3**	**CaO**	**MgO**	**Na_2O**	**Cr_2O_3**	**SiO_2**	**FeO**	**CaF_2**
32	57.36	28.64	2.15	4.58	4.12	1.85	1.30	0
33	55.10	27.55	2.65	4.90	4.10	2.15	1.55	2
34	54.18	27.10	2.34	4.85	4.15	1.95	1.43	4
35	53.00	26.50	2.20	4.80	4.10	1.92	1.48	6
36	51.07	25.53	2.35	4.75	4.20	2.10	2.00	8
37	50.00	25.00	2.33	4.82	4.18	1.95	1.72	10

Table 7. Compositional details of slags. (simultaneous variation of MgO and CaF_2)

Slag No.	Al_2O_3	CaO	MgO	Na_2O	Cr_2O_3	SiO_2	CaF_2	Al_2O_3/CaO
38	60.0	24.0	4.0	5.0	4.0	3.0	0.0	2.5
39	58.6	23.4	4.0	5.0	4.0	3.0	2.0	2.5
40	57.1	22.9	4.0	5.0	4.0	3.0	4.0	2.5
41	55.7	22.3	4.0	5.0	4.0	3.0	6.0	2.5
42	54.3	21.7	4.0	5.0	4.0	3.0	8.0	2.5
43	58.6	23.4	6.0	5.0	4.0	3.0	0.0	2.5
44	57.1	22.9	6.0	5.0	4.0	3.0	2.0	2.5
45	55.7	22.3	6.0	5.0	4.0	3.0	4.0	2.5
46	54.3	21.7	6.0	5.0	4.0	3.0	6.0	2.5
47	53.0	21.0	6.0	5.0	4.0	3.0	8.0	2.5
48	51.4	22.9	8.0	5.0	4.0	3.0	0.0	2.5
49	55.7	22.3	8.0	5.0	4.0	3.0	2.0	2.5
50	54.3	21.7	8.0	5.0	4.0	3.0	4.0	2.5
51	53.0	21.0	8.0	5.0	4.0	3.0	6.0	2.5
52	51.4	20.6	8.0	5.0	4.0	3.0	8.0	2.5
53	63.0	21.0	4.0	5.0	4.0	3.0	0.0	3.0
54	61.5	20.5	4.0	5.0	4.0	3.0	2.0	3.0
55	60.0	20.0	4.0	5.0	4.0	3.0	4.0	3.0
56	58.5	19.5	4.0	5.0	4.0	3.0	6.0	3.0
57	57.0	19.0	4.0	5.0	4.0	3.0	8.0	3.0
58	61.5	20.5	6.0	5.0	4.0	3.0	0.0	3.0
59	60.0	20.0	6.0	5.0	4.0	3.0	2.0	3.0
60	58.5	19.5	6.0	5.0	4.0	3.0	4.0	3.0
61	57.0	19.0	6.0	5.0	4.0	3.0	6.0	3.0
62	55.5	18.5	6.0	5.0	4.0	3.0	8.0	3.0
63	60.0	20.0	8.0	5.0	4.0	3.0	0.0	3.0
64	58.5	19.5	8.0	5.0	4.0	3.0	2.0	3.0
65	57.0	19.0	8.0	5.0	4.0	3.0	4.0	3.0
66	55.5	18.5	8.0	5.0	4.0	3.0	6.0	3.0
67	54.0	18.0	8.0	5.0	4.0	3.0	8.0	3.0

In the second stage, 30 nos of slags were prepared, with composition of the slags in line with the composition of slags encountered in industry to study the effect of simultaneous variation of CaF_2 and MgO. For the first 15 nos of these slags (slag no. 38 to slag no. 52) the Al_2O_3/CaO ratio was fixed at 2.5 and for the rest 15 nos of slags (slag no. 53 to slag no. 67) the ratio was fixed at 3.0. For all these slags the Na_2O, Cr_2O_3, SiO_2 contents were respectively fixed at 5.0 wt%; 4.0 and 3.0 wt%, CaF_2 varying from 0 to 8.0 wt% with simultaneous variation of MgO from 4.0 to 8.0 wt%. The compositional details are presented in **Table 7**.



1. Cooling water tank.
2. Cooling water recirculating tank.
3 Light source
4. Regulating transformer for light source.
5. Optical Bench.
6 High temperature electrical furnace with Speciman carriage.
7. Observation and photo microscope.
8. Digital thermometer.
9. Regulating transformer for high temperature electrical furnace.

Fig. 1. Schematic Diagram of the Heating-microscope.

2.2 Experimental Set-up and Reporting of the Experimental Data

The Heating Microscope method is adopted for recording the characteristic temperatures. Schematic diagram of the instrument is presented in **Fig. 1**. A detailed description of the instrument and its working principles is presented else where.[14)]

The sample, in the form of a 3 mm cube, is heated in an electric furnace in the microscope assembly. The shape change of the sample as a result of heating is photographed by a camera. A grid-division which is simultaneously photographed with the sample and the temperature to which the sample is being heated facilitate identification of the four characteristic temperatures. The four temperatures are reported as follows by following the German Industrial Standard 51730.

IDT : The temperature at which the first rounding up of the cube edges takes place.

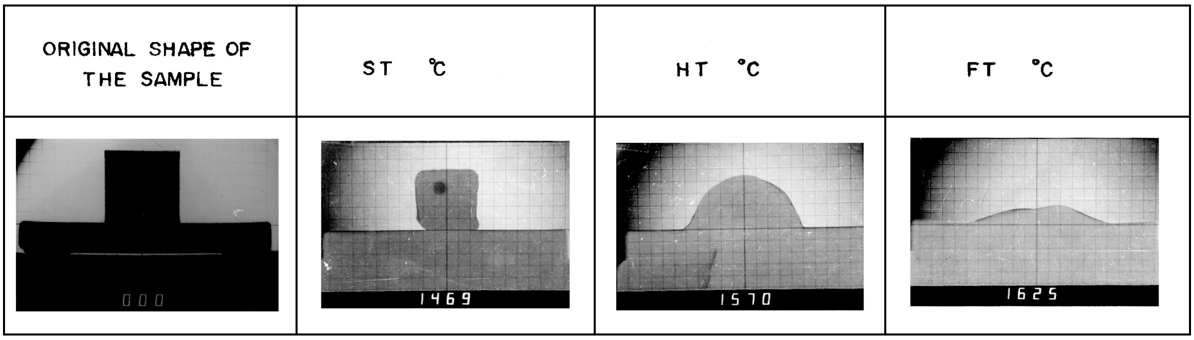


Fig. 2. Micro Photographs illustrating the characteristic Temperatures of a slag sample. (Slag No. 3)

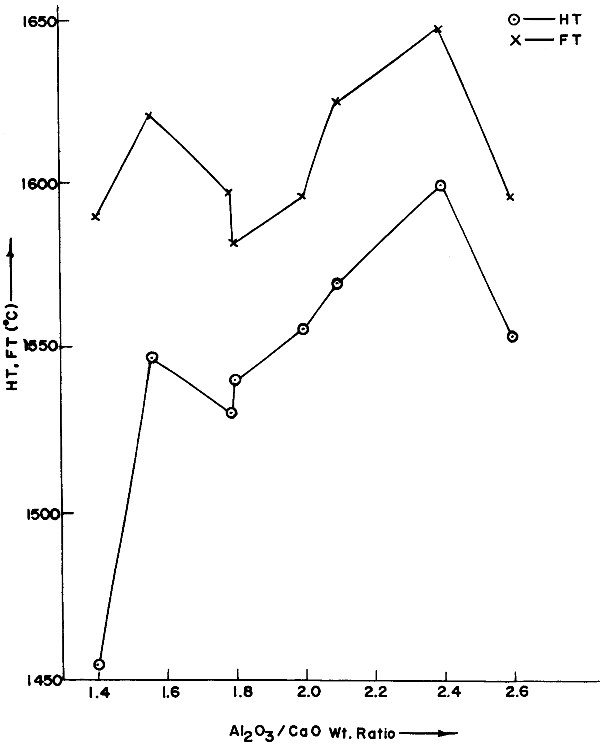


Fig. 3. Relationship between HT, FT and Al_2O_3/CaO wt. ratio.

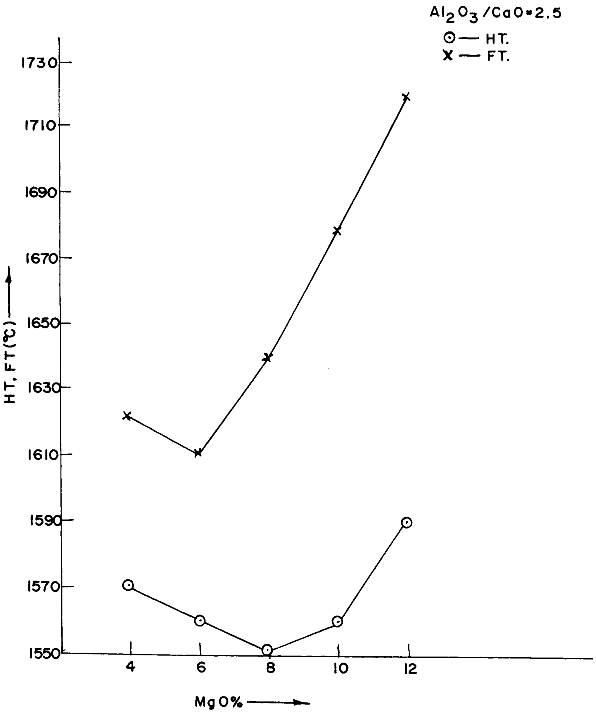


Fig. 4. Relationship between HT, FT and wt% MgO.

ST : The temperature at which the outline of the cube shape starts changing and is measured when the specimen sinks by one division.

HT : The temperature at which the specimen assumes hemispherical shape and is measured as the temperature at which the height of the specimen is equal to half of its base-length.

FT : The temperature at which the height of the specimen is equal to one-third of the height it had attained at HT.

The micro-photographs of one of the samples examined are presented in **Fig. 2** as an illustration.

The rate of rise of temperature is kept at 100°C per minute maximum up to the first 10 000°C after which it is kept at 40 to 60°C per minute, as heating rate faster than this suppresses the action of charge and hence elevates the characteristic temperatures, especially HT & FT.

Two random samples are collected per slag for examinations. The average of the two readings is reported in case the two differ by not more than 50°C. In case the difference is more than 50°C fresh samples are drawn and the experiment is repeated.

3. Results and Discussions

The effect of Al_2O_3/CaO ratio, MgO content and CaF_2 content on the HT and the FT, the two important characteristic temperatures which greatly influence the Aluminothermic reduction process, have been presented in **Figs. 3** through **6** for the first stage of 37 nos. of slags. In a similar way the results of the second phase of 30 nos. of slags pertaining to the tests for simultaneous variation of MgO and CaF_2 at two different Al_2O_3/CaO ratios are presented in **Fig. 7** through **9**.

3.1. Variation of Al_2O_3/CaO Rratio

The binary Al_2O_3–CaO diagram reveals that CaO additions bring down the melting temperature of Al_2O_3 considerably. However, the aluminothermic slags, in addition to having Al_2O_3 and CaO as major constituents also contain other oxides like SiO_2, MgO, Na_2O, FeO, *etc.* Thus it is necessary to investigate how these oxides combindly influence the liquidus temperature of these slags at various Al_2O_3/

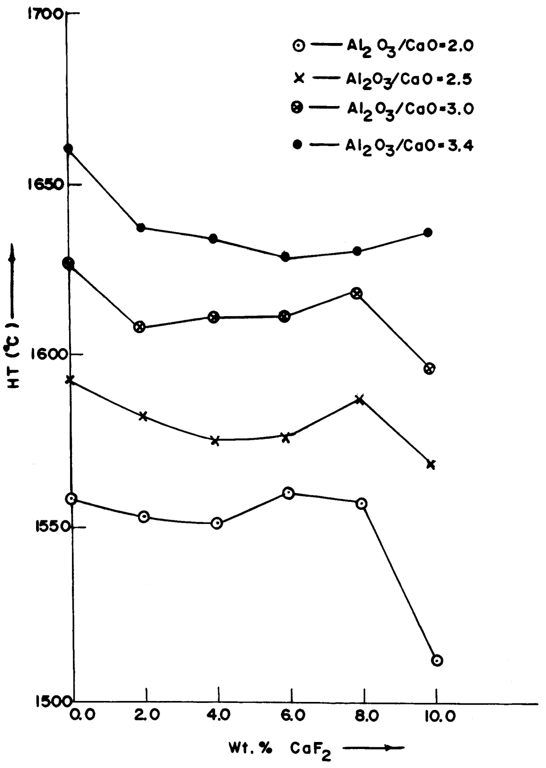


Fig. 5. Relationship between HT and wt% CaF_2 at different Al_2O_3/CaO wt. ratios.

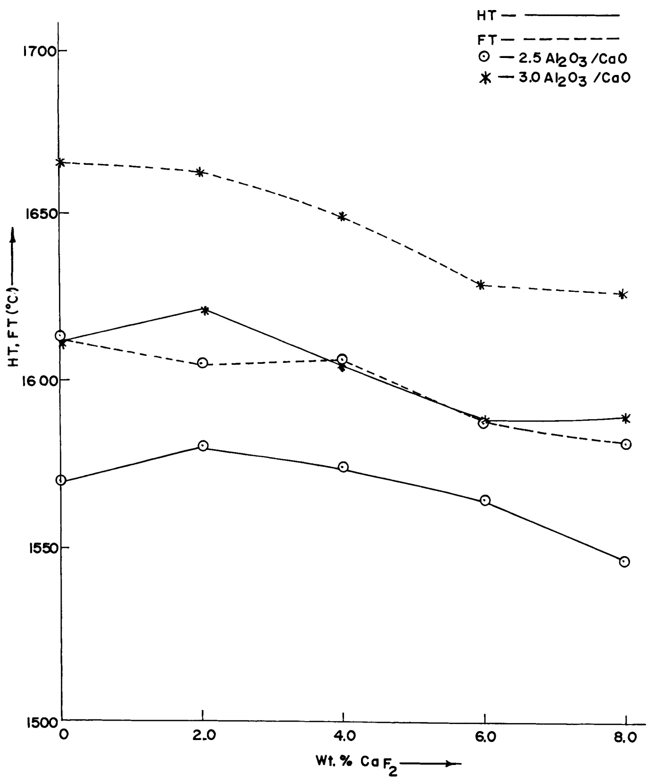


Fig. 7. Relationship between HT, FT and wt% CaF_2 at different Al_2O_3/CaO wt. ratio (wt% MgO=4.0)

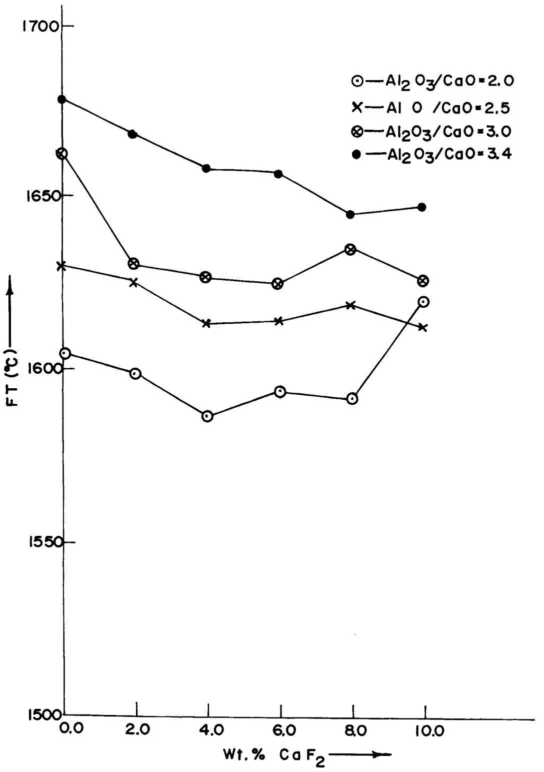


Fig. 6. Relationship between FT and wt% CaF_2 at different Al_2O_3/CaO wt. ratios.

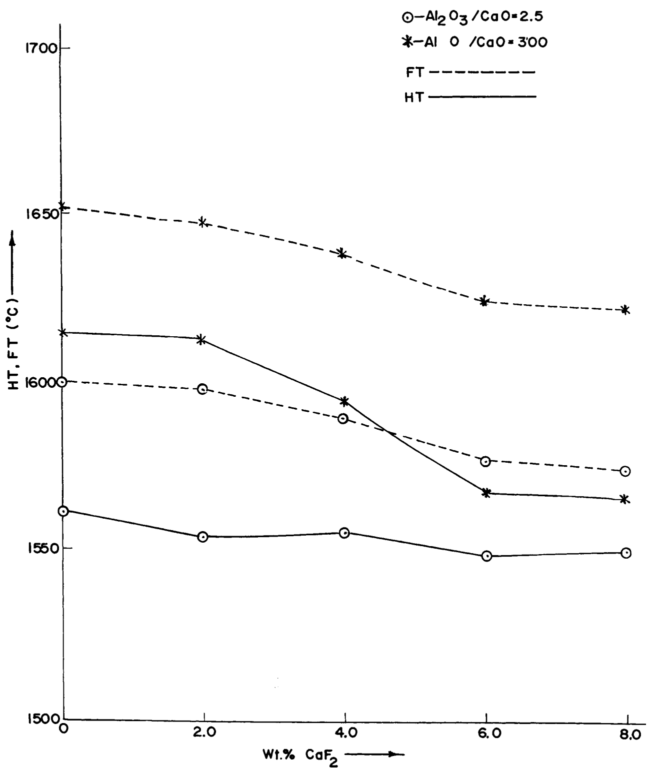


Fig. 8. Relationship between HT, FT and wt% CaF_2 at different Al_2O_3/CaO wt. ratio (wt% MgO=6.0)

CaO ratios and hence influence the resultant slag characteristics.

As illustrated in Fig. 3 there is no regular trend of variation of HT and FT with increase of Al_2O_3/CaO ratio specially in the presence of other oxides amounting to a total of 13 to 20%. At lower values of the ratio both the HT and the FT values are comparatively low.

Nityananda and Fine[5] and Nurse *et al.*[15] made liquidus temperature measurements of Al_2O_3–CaO system. They reported a minimum of liquidus temperature at Al_2O_3/CaO

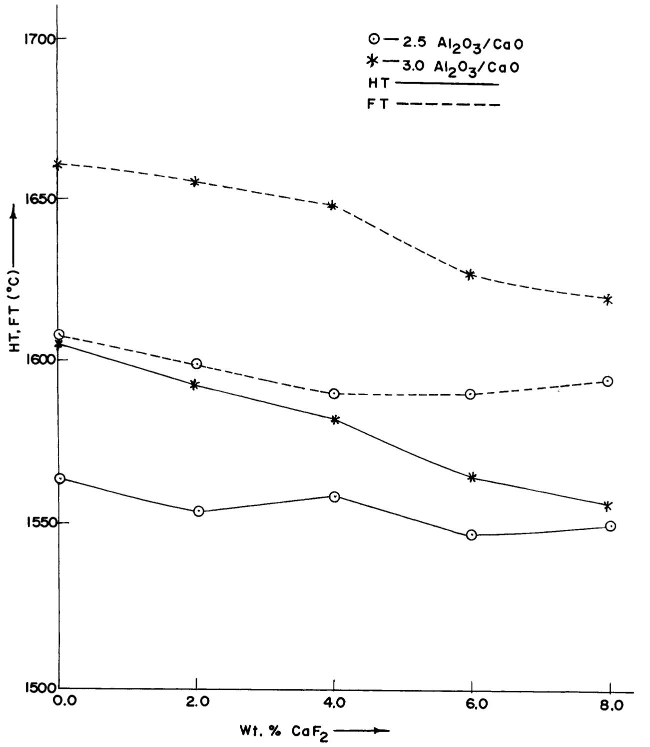


Fig. 9. Relationship between HT, FT and wt% CaF_2 at different Al_2O_3/CaO wt. ratio (wt% MgO=8.0)

ratio of about 1.0, indicating that the same increased rapidly with increase in the ratio. As the ratio in the present investigation is much above 1.0, the HT is found to be much higher than the values they have reported. It is further noticed from the investigation of Nityananda and Fine[5] that in the presence of more than 5% TiO_2 the effect of Al_2O_3/CaO is erratic. In the present investigation a similar nature of curves for both HT and FT have been observed within the range of Al_2O_3/CaO ratio from 1.4 to 2.6 in the presence of other oxides such as SiO_2, MgO, FeO, Cr_2O_3 *etc.* It may be observed from Fig. 3 that the nature of variation of FT is similar to that of HT except in the range of Al_2O_3/CaO ratio of 1.79 to 1.80 where the former shows a decrease while the later shows an increase. The non-regular variation may be due to the interaction of various constituents in different ways at different temperatures and formation of various types of flow units. Hence, from the extraction process point of view slag no.8 with Al_2O_3/CaO ratio of 1.4 is the best as it gives the lowest HT. However, this slag is a long slag with the difference between FT and HT being more than 50°C (136°C to; be precise). It is interesting to note that the difference between the HT and the FT continuously decreases with the increase of Al_2O_3/CaO ratio from 1.4 to 2.0, as the HT increases faster than FT with increase in the ratio.

In metallothermic processes the fusion point of the slag should be low and the difference between FT and HT should be less. This will result in an enhanced mobility of the slag and hence ensure better slag metal separation. This fact should be kept in mind while deciding the flux additions for better yield of metal.

3.2. Variation of MgO Content

The effect of MgO content on the fusion behaviour of ferrochrome slags at Al_2O_3/CaO ratio of 2.5 is presented in Fig. 4. It was decided to examine if replacement of the basic constituent CaO by another basic oxide MgO could improve the slag characteristic at high Al_2O_3/CaO ratio. It is seen that although the increase of MgO content in the range of 4 to 8% decreased the liquidus temperature, beyond 8.0 wt% MgO the trend is reversed. This may be due to very high melting point of MgO in the order of 2 800°C. Even the minimum liquidus temperature is found to be higher than that obtained at Al_2O_3/CaO ratio of 1.4 to 1.8. However, the trend is similar to what is observed in the case of Blast furnace slags within the range of MgO content from 0–16% as reported by Athappan[16] and 0–30% MgO as reported by Singh *et al.*[17]

Another interesting feature observed is that the difference between FT and HT continuously goes on increasing with the increase in the MgO content and that the slag becomes a long-slag (FT-HT is above 50°C) beyond 6.0 wt% MgO additions. Hence, it is concluded that so far as melting is concerned, higher MgO content (beyond 6.0 wt%) at Al_2O_3/CaO ratio of 2.5 is not beneficial. However, further investigations may have to be carried out at various other values of the Al_2O_3/CaO ratio before reaching at any decisive conclusion.

3.3 Variation of CaF_2 Content

Calcium fluoride is known to be a good fluxing agent and more as a reagent resulting in a more fluid slag. Hence, it was decided in the course of the present investigations to replace part of the CaO by CaF_2.

The nature of variations of HT and FT with increase in CaF_2 content from 0–10% at four different Al_2O_3/CaO ratios are shown in Figs. 5 and 6 respectively.

It is observed from Fig. 5 that the nature of variation of HT with increase in CaF_2 content is almost same at different Al_2O_3/CaO ratios exhibiting a decreasing trend initially but showing slight increase at higher CaF_2 contents. It is interesting to see that for all fluoride containing slags at the four Al_2O_3/CaO ratios investigated, the minimum HT occurs in the range of 4–6 wt% CaF_2 content, except slag no. 35. It is further observed that the HT increases with the increase of Al_2O_3/CaO ratio at any given wt. percentage of CaF_2. Hence, from the process point of view, slags with low Al_2O_3/CaO ratio and CaF_2 contents within 4.0 to 6.0 wt% are more acceptable.

Figure 6 shows the variation of the FT with increase of CaF_2 content at the four Al_2O_3/CaO ratios. The variation shows a similar trend. It is further observed that as in case of HT the lower values of FT are obtained in the range of 4.0 to 6.0 wt% of CaF_2 content at all the Al_2O_3/CaO ratios except at 3.4 where higher CaF_2 content result in lower values of FT. Hence, at higher Al_2O_3/CaO ratios CaF_2 seems to be an effective fluxing constituent.

Increase of CaF_2 is expected to decrease the liquidus temperature as reported for CaF_2–Al_2O_3–CaO ternary system[18] due to the formation of various types of calcium Aluminates like, $CaO \cdot Al_2O_3$, $3CaO \cdot Al_2O_3$, $12CaO \cdot 7Al_2O_3$,[19–21] but the results presented in Figs. 5 and 6 show that the decrease is not smooth. This may be due to the fact that there is some compositional change as CaF_2 reacts with Al_2O_3 to form volatile AlF_3 thus depleting the slag of some

CaF_2. Hence, when the Al_2O_3/CaO ratios are higher, the CaF_2 can be used as an effective flux to bring down the HT and the FT substantially. This will ensure high metal recovery at low energy expenditure.

3.4. Simulataneous Variaton of CaF_2 and MgO Contents

The results of variation of CaF_2 from 0 to 8.0 wt% and MgO from 4.0 to 8.0 wt% at two different ratios of Al_2O_3/CaO at 2.5 and 3.0 are presented in Figs. 7, 8 and 9 at 4.0, 6.0 and 8.0 wt% MgO content respectively.

The decrease of HT and FT are however smooth here in the presence of higher percentages of MgO. It is possible that MgO will react to some extent with CaF_2 to form MgF_2 and CaO. The liquidus temperature of CaF_2–Al_2O_3–MgO system has been reported by Salt.[22] The measured values agree with the liquidus ranges reported by him, within the composition range. The liquidus temperatures at Al_2O_3/CaO ratio of 2.5 are found to be in the range of 1 545–1 570°C where as the same value at low MgO contents range between 1 569–1 592°C. Similar lower results are obtained at Al_2O_3/CaO ratios of 3.0 also.

4. Conclusions

The following conclusions are drawn from the present investigation.

(1) Both the liquidus temperature (represented as HT) and the flow temperature (represented as FT) are low at lower Al_2O_3/CaO ratios in the presence of other oxides such as SiO_2, MgO, Na_2O, FeO, *etc.* taken together, amounting to 15.0 wt%.

(2) The difference between HT and FT decreases continuously with increase in Al_2O_3/CaO ratio in the range of compositions of slags mentioned above.

(3) At Al_2O_3/CaO ratio of 2.5, MgO to an extent of 8.0 wt% is beneficial in reducing the liquidus temperature.

(4) CaF_2 to an extent of 4.0 to 6.0 wt% acts as a powerful flux to reduce both FT and HT at all values of Al_2O_3/CaO ratios, more so at higher values of the ratio.

(5) Simultaneous variation of MgO and CaF_2 gives a regular trend of lowering of FT and HT values and to that extent high percentage of MgO is effective in the presence of CaF_2.

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