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A Study on Crystallinity of Mold Fluxes for Continuous Casting of Peritectic Steel

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Abstract

Over 90% of the steel produced in the world is cast via continuous casting route. Bulk of this steel, belongs to peritectic grade having carbon content ranging between 0.1 to 0.5 %. The peritectic reaction is associated with volume contraction during α -ferrite to austenite transformation which is considered to be the root cause of the stresses evolved during solidification of casting leading to the appearance of longitudinal cracks, which is a matter of concern.

It is known that the controlled rate of horizontal heat transfer minimizes the thickness of steel shell which is able to bear contraction stresses, reducing thereby the tendency of formation longitudinal crack. This is achieved by allowing to form thicker layer of solidified flux, between the mold and the strand, with higher fraction of crystalline phase.

In the light of this, X-Ray Diffraction and Metallographic techniques were established to determine fraction of crystalline phase in fluxes. The fluxes were decarbed, melted and quickly solidified. Due to fast cooling samples were almost in glassy state. Glassy to crystalline transformation was then carried out isothermally at temperatures between 700°C to 1000°C and for a time period of 2 to 6 hrs. Fraction of crystalline phase was determined in all the three fluxes as a function of temperature and time.

Based on these studies, attempt has been made to explain the behavior of the fluxes in the context of formation of longitudinal cracks.

Keywords: Crystallinity, mold fluxes, peritectic steel Cuspidine phase.

Introduction

Continuous casting mold fluxes play an important role in the success of concast process. Mold fluxes are basically synthetic slags, belong to the system CaO- SiO₂- Al₂O₃- Na₂O - CaF₂ and are used in the powder form to cover the molten steel in water cooled copper mold, in order to protect the steel from reoxidation and also to act as a thermal insulator. Much more important, that it also serves as a lubricant between the mold and the strand and regulate the horizontal rate of heat transfer.

Experimental

This chapter describes experimental methods employed in the present work.

Composition And Properties Of Flux Samples

Three powder samples were taken from powder manufacturing company in Pune. Out of these three samples only one sample gave defect free casting while casting peritectic steel.

In plant trial, these samples were taken on the basis of their chemical composition, melting

point, basicity, viscosity and crystallization temperature etc. out of these three samples one sample T1 was having high basicity and high crystallization temperature, and the other sample T2 was having low basicity and low crystallization temperature, both are at extreme end, but the third powder T-A was having basicity and crystallization temperature in between these two samples, and only this powder gave defect free cast.

Chemical compositions as well as properties of all these three samples are given in table 2.1.

Sample Preparation

Decarburization Of Flux

Flux powders are mainly mixtures of various compounds in right proportion of minerals such as feldspar, calcium carbonate, powdered glass, calcium fluoride plus free carbon up to 10%. About 100g of the three powders samples T-1, T-A and T-2 were taken. The flux powder contains some amount of free carbon in various forms as per its known composition. This carbon poses problem during

melting of this powder. Thus it was necessary to remove the carbon before it is subjected to melting. It was carried out by heating the powders in alumina crucibles at 800°C for 8 to 10 hrs in muffle furnace and which was followed by furnace cooling. After bringing its temperature to room temperature the powder colour became white. In order to ensure that the samples were completely decarbed, one representative sample was tested for carbon in C and S apparatus which confirmed absence of any leftover carbon. This powder then taken out from the alumina crucible and was kept in plastic bags and kept away from air in order to keep the same moisture free. Now the powder was ready for melting

Table: 2.1 Chemical analysis and physical properties of casting fluxes T-1, T-A & T-2

Chemical analysis	% of T-1	% of T-A	% of T-2
SiO ₂	19.90	26.7	33.70
CaO	41.10	32.1	25.90
MgO	3.30	0.2	4.20
Al ₂ O ₃	7.90	5.4	4.70
TiO ₂	0.0	0.3	0.0
Fe ₂ O ₃	2.40	1.2	1.50
MnO ₂	0.0	1.6	0.10
P ₂ O ₅	0.0	0.0	0.0
Na ₂ O	7.90	7.1	11.10
K ₂ O	0.40	0.2	0.20
F	7.90	7.2	8.90
Li ₂ O	-	-	-
B ₂ O ₃	0.70	-	0.70
H ₂ O	0.11	0.2	0.20
CO ₂	6.5	11.5	7.30
C _{tot}	3.90	7.4	5.50
C _{free}	2.20	4.3	3.50

Physical properties	T-1	T-A	T-2
Basicity, %	2.06	1.20	0.77
Crystallization temp, °C	1219	1140	1054
Melting Point, °C	1070	1125	1030
Bulk density, Kg/dm ³	0.70	0.59	0.70
Viscosity, Pa.s		0.09	
Softening Point, °C		1090	

Melting And Casting Of Flux

As the melting point of these flux powders is high around 1000-1200°C, induction furnace was used for melting. In view of the fact that the graphite was used as a crucible material and the total material to be melted was hardly about 50g or so, the total time required for melting or to raise the temperature up to 1400°C, was of about 5 minutes or so. It was apprehended that carbon of the graphite crucible

might have not reacted with the flux material which was composed of mainly oxides.

In such short time the crucible was then kept in the induction coil of the furnace and power was applied so that heating would take a place. When the crucible became sufficiently hot the temperature of it was measured by inserting a platinum-platinum-13% rhodium thermocouple connected to multimeter. During temperature measurement induction was stopped for few seconds in order to avoid induction effect on the wires of T.C. Reading on the multimeter was then noted and corresponding temperature was found out from the standard charts by adding the room temperature. When it came around 1346°C to 1400°C, then heating was stopped and the red hot crucible was taken out instantly from the coil and the molten flux was poured into the steel moulds for casting.

The mold is also provided with a top plate which was also made up from the same material and having the same size. Just after pouring the molten flux in mold, top plate was placed on it in order to obtain samples having uniform thickness of 2 to 3mm. earlier it was thought to poured the molten flux in a cavity formed between a cope and drag type of mold. However, it was found difficult to pour the small quantity of 50g through a narrow passage of the pouring sprue without loss of its temperature. Furthermore molten flux did not wet stainless steel mold surface.

The stainless steel was chosen as mold material in place of copper because it could withstand high temperature of 1400°C.

Initially 12 separate molds of 10mm inner diameter and 20mm outer diameter and of inside depth of 2mm were made from stainless steel rod. However due to non-wetting character of molten flux with stainless steel, it was not possible to obtain 2 to 3mm thin casting, which is usual thickness of the solidified flux between the mold and the steel strand.

A separate mold and its top plate were fabricated with the help of Auto Industries, Pune, from stainless steel. Schematic diagram of the same is shown in Figure 2.3 and 2.4. It was made up from 304 stainless steel and machined by VMC machine and was of size 140mm length X 54mm width X 8mm thick. With the help of this it was possible to cast 24 specimens of 2 to 3mm thickness and of 15mm diameter at a time from one single melt.

Before pouring, graphite powder was sprinkled over these moulds in order to avoid sticking of the sample with the mould. Total 24 samples were cast and after its solidification the samples were immediately quenched in the water bath. Once the sample was immersed in water it had detached from the mould automatically.



Figure 2.1: Photograph of the induction melting unit.

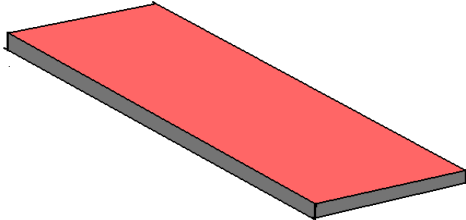


Figure 2.2: stainless steel top plate

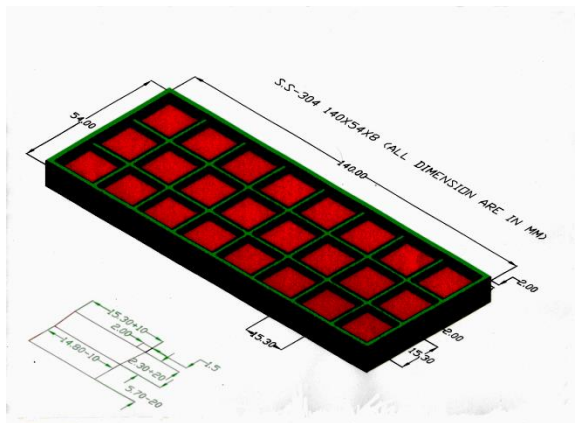


Figure 2.3: Stainless steel mold

Heat Treatment Of Flux Samples After Casting

STRESS RELIEVING

HEAT TREATING AT 700°C, 800°C, 900°C & 1000°C

MUFFLE FURNACE

Techniques Employed For X-Ray Diffraction Study

X-Ray Diffraction Technique For Quantitative Analysis Of Phases

X-Ray Diffraction has been used for quantitative phase analysis as being used for phase quantification since 1936. In this work internal standard method was used to calculate weight fraction of cuspidine phase. In this method a diffraction line from the phase to be determined is compared with a line from the standard substance mixed with the sample in known quantities. The internal standard is therefore restricted to sample in powder form. Internal standard is used for the analysis of cement and the analysis of clay

minerals. In any method involving powders, accurate sampling and homogeneous mixing can present problems. They are not trivial, and errors in these operations can produce large errors in the final results [7].

Crushing and Mixing Of Standard For XRD

The samples were prepared for XRD in powder form, therefore the samples were crushed to fine size in **pestle and mortar** by packing them in plastic paper to avoid flying of the pieces during hammering, and made it more fine. In order to match the particle size and size distribution of the powder with the powder of α - Alumina.

The powders (prepared mould powder and α - Alumina) were then mixed, and the mixing process was also carried out in **pestle and mortar** where α - Alumina was added as external indicator for powder method of X-ray diffraction (XRD). These powders were mixed in the proportion of 9:1 by weight where 9 parts were of mould powder and 1 part of external indicator powder (α - Alumina), and prepared total 1gm sample for XRD.

Hayashi et al [2] also determined fraction of crystalline phase by employing Mn_2O_3 as an external standard. X-Ray diffraction of Mn_2O_3 does not overlap with any of the lines of the compounds such as cuspidine ($3CaO \cdot 2SiO_2 \cdot CaF_2$), nepheline ($NaAlSi_3O_8$), wollastonite ($CaO \cdot SiO_2$) etc. which are commonly present in majority of fluxes however Mn_2O_3 was not available in small quantities and found to be expensive. Many other scientists have used α -Alumina as an external standard, as it is also very stable. Only drawback of using α - Alumina is that its X-Ray pattern may interfere with the Al_2O_3 already present in the fluxes. Eventually it was decided to use α - Alumina as an external indicator [4].

Techniques Employed For Metallographic Study

The samples of present work were basically molten oxides composed of glassy and/or crystalline phases. Methods employed to obtain microscopic structures of such samples are explained in this section.

Preparation Of Samples For Metallography

After casting heat treatment was given to the flux samples as discussed in section 2.3. The as cast and stress relieved specimens are subjected to following processes in order to determine their microscopic structures .

A) Cold mounting

The as cast and stress relieved samples were brittle, thin and highly breakable therefore they were first cold mounted by using cold mounting powder and liquid. For cold mounting samples were placed in mold and slurry of cold mounting powder and liquid was poured in it. After few minutes it got solidified,

after solidification sample was removed from mold. Same procedure was repeat for all samples.

B) Polishing

All these cold mounted samples then polished to obtain mirror like surface. It was done by sequence of operations i.e. first polished on belt grinder then on emery paper and finally on lap wheel with diamond paste. It uses the abrasive particles that are not fixed, but suspended in a liquid among fibers of cloth. The objective was to produce bright mirror like or reflecting or refracting surface commonly referred to as a polished surface.

Results & Discussions Of Xrd & Metallograpy

Three different continuous casting mold fluxes manufactured by company in Pune were obtained In order to study the effect of isothermal heat treatment on crystallinity as a function of time. As explained earlier one of these fluxes namely T-A was found to give satisfactory results vis a vis longitudinal cracking defect observed in peritectic grade billets. The other two samples were chosen on the basis of their extreme basicity and viscosity values for the sake of comparison of crystallinity of fluxes.

As mentioned earlier in section 2.4 the samples were decarbed and melted in high frequency in Induction furnace in the laboratory and then subjected to isothermal phase transformation from amorphous to crystalline state at temperatures 700°C, 800°C, 900°C, 1000°C and holding the same at these temperatures with varying time from 2hrs to 6hrs respectively

The extent of crystallization taken place during these experiments and identification of crystalline phases was ascertained by X-Ray Diffraction quantitative phase analysis technique and Metallography. Results thus obtained are given in section 3.1 and 3.2 respectively.

Phase Identification & Quantitative Analysis By X-Ray Diffraction Technique

As mentioned earlier, in section 1.4 X-Ray Diffraction patterns were obtained by using XRD unit of the Dept. of Physics, University of Pune. In all 24 samples (8 samples per mold flux) were prepared In order to obtain X-Ray Diffraction patterns. Diffraction patterns thus obtained are given in figures from Figure 3.1 to Figure 3.9 (These are for sample T-A). The experimental condition of each specimen is given namely flux sample number, temperature of the heat treatment and the holding time respectively along with the X-Ray Diffraction pattern. Figure 3.1 to 3.7 are of specimens which are used in powder form along with the external standard of α -Alumina,

However Figure 3.8 and Figure 3.9 are X-Ray Diffraction patterns of the sample T-A retrieved specimen from the meniscus as such in the solid form. Since X-Ray penetrates hardly of micron depth, two X-Ray Diffraction patterns were obtained, one from the copper mold and the other from hot strand side.

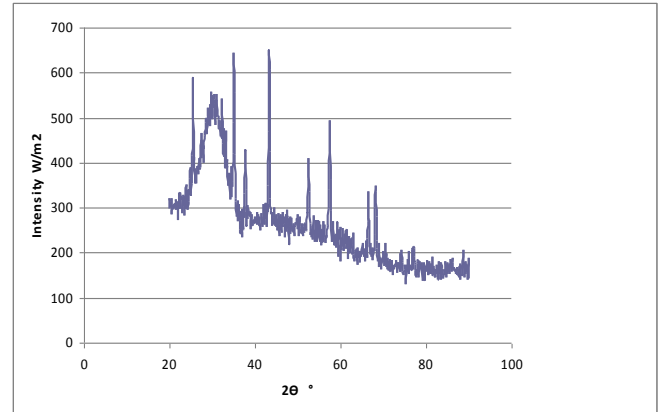


Fig. 3.1: Sample T-A - As cast and stress relieved at 600°C

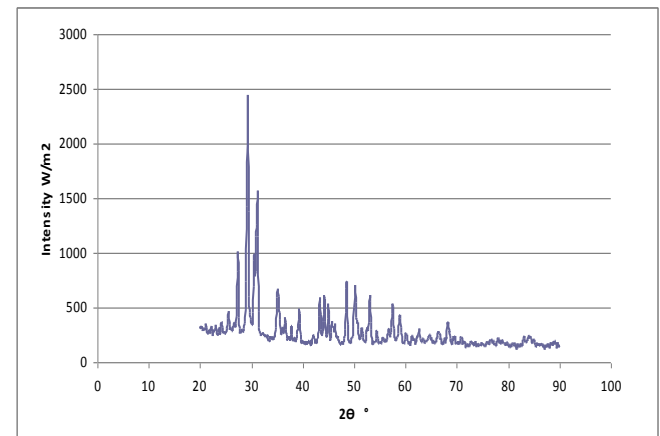


Figure 3.2: Sample T-A - Heat treat temp. 800°C - Holding time 2hrs

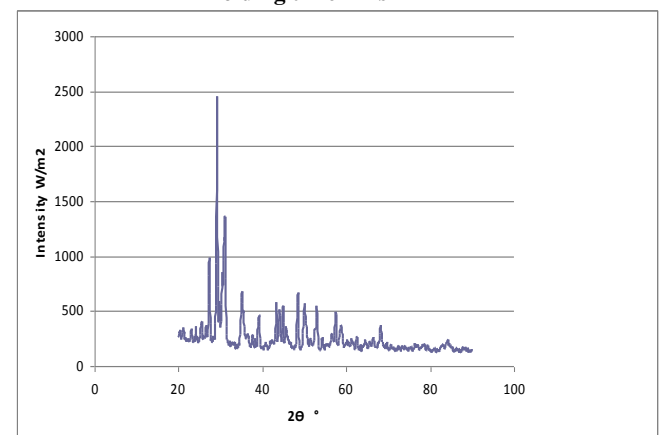


Figure 3.3: sample T-A – Heat treat temp. 800°C – Holding time 6hrs

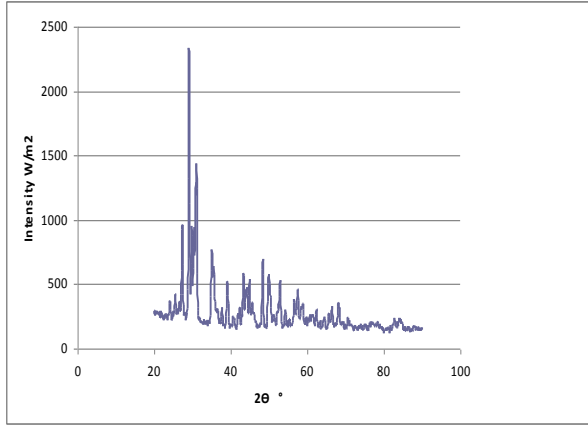


Figure 3.4: Sample T-A – Heat treat temp. 900°C – Holding time 2hrs

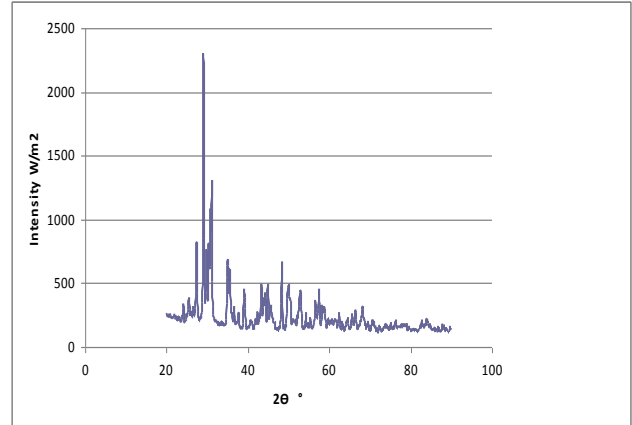


Figure 3.7: Sample T-A – Heat treat temp. 1000°C – Holding time 6hrs

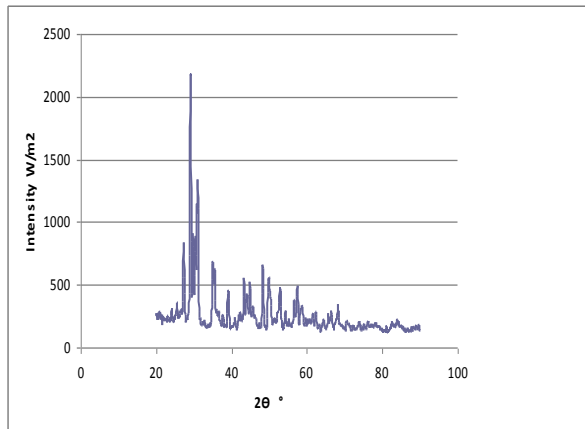


Figure 3.5: Sample T-A – Heat treat temp. 900°C – Holding time 6hrs

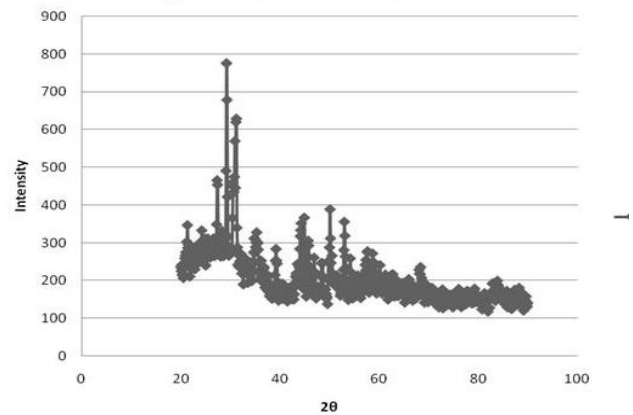


Figure 3.8 Sample T-A - retrieved from meniscus – Copper mold side

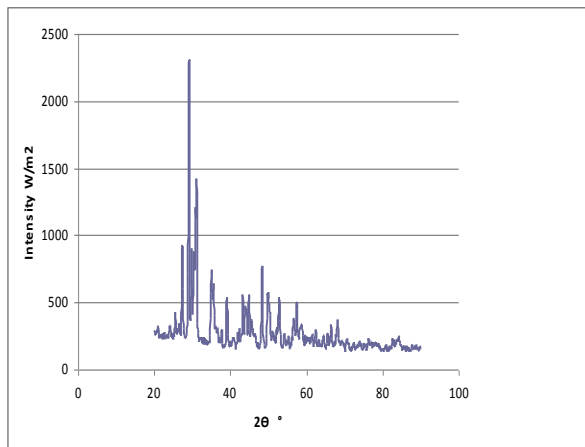


Figure 3.6: Sample T-A – Heat treat temp. 1000°C – Holding time 2hrs

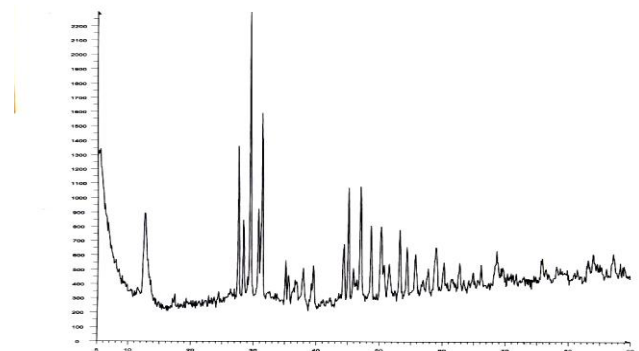


Figure 3.9 Sample T-A - Retrieved from meniscus – Hot strand side

Data Obtained From X-Ray Diffraction Graphs On Crystallinity

As cast and stress relieved samples of all the compositions have formed blurred XRD graphs, similar to a typical graph of an amorphous phase. This is mainly true for T-A and T-2 samples. The mold side retrieved sample shows this effect much more emphatically, figure 3.8 However, few peaks not so sharp, can be seen in these graphs also which corresponds to crystalline phases.

Comparing with this, as cast and stress relieved sample shows some amount of crystallinity and this effect was found on much higher side in case of T-1 sample which was obtained from flux with higher basicity. It is concluded that even at a low temperature of 600°C, stress relieved treatment has also induced crystallinity in all these samples and this phenomena is promoted further by high basicity.

With the aid of PCPDF software different crystalline phases were identified at different peaks seen at various XRD angles which is shown in table 3.1. As a matter of fact every phase, there are large number of XRD angles at which peaks are also seen. However the taller or predominant is considered, as it is a commonly practised, for identification of a phase.

The peak of cuspidine was most predominant in all the heat treated samples. With the help of the said software it was possible to determine the intensity of given peak. Furthermore by comparing this intensities with that of the external standard of α- Alumina, by employing the following formula % of each crystalline phase of cuspidine, nepheline and wollastanite was calculated. V is.

$$(Wt \% \text{ CUSPIDINE}) / (Wt \% \alpha\text{-ALUMINA}) = [(I_{MAX \text{ for CUSPIDINE}}) / (I_{MAX \text{ FOR } \alpha\text{-ALUMINA}})] * 100 \quad [7]$$

The data thus calculated is given in table 3.2

Table 3.1 XRD angle 2θ versus type of crystalline phase

Sr. No.	Angle at which tallest peak is seen, 2θ°	Crystalline phase
1.	29.1	Cuspidine (3CaO 2SiO ₂ CaF ₂)
2.	29.8	Nepheline (NaAlSiO ₄)
3.	23.1	Wollastanite (CaO SiO ₂)
4.	35.1	α – Alumina, external indicator

Table: 3.1 A % crystalline phases in as cast and heat treated samples of T-1 as a function of temp. and holding time

Sr. No	Heat treating temp. °C	Holding time hrs	% Cuspidine	% Nepheline	% Wollastanite
1	600	1	15.6	4.8	6.1
2	800	2	35.6	5.0	7.3
3	800	6	38.1	5.6	7.4

4	900	2	36.3	5.5	6.7
5	900	6	35.4	5.4	6.9
6	1000	2	39.0	6.4	7.0
7	1000	6	39.3	5.6	6.5

Table: 3.1 B % crystalline phases in as cast and heat treated samples of T-A as a function of temp. and holding time

Sr. No	Heat treating temp. °C	Holding time hrs	% Cuspidine	% Nepheline	% Wollastanite
1	600	1	8.09	7.6	5.5
2	800	2	21.0	6.1	5.7
3	800	6	29.02	9.4	5.4
4	900	2	30.5	12.3	3.4
5	900	6	31.9	13.5	3.6
6	1000	2	30.0	12.0	3.3
7	1000	6	33.07	11.0	3.0
8	-	1	25.7	-	-

Table: 3.1 C % crystalline phases in as cast and heat treated samples of T-2 as a function of temp. and holding time

Sr. No	Heat treating temp. °C	Holding time hrs	% Cuspidine	% Nepheline	% Wollastanite
1	600	1	9.56	6.6	4.3
2	700	2	19	13.0	5.6
3	700	6	19.3	12.1	7.0
4	800	2	22.6	6.6	5.0
5	800	6	23.1	9.1	6.5
6	900	2	28	5.1	4.3
7	900	6	26.5	4.5	4.3

Phase Identification & Quantitative Analysis By Metallography

As mentioned earlier, in section 2.5 samples were prepared for Metallography and images were obtained by using Image Analyzer at 100X magnification. All samples were prepared in order to obtain Metallography images. Metallography images thus obtained are given in figures from Figure 3.10 to Figure 3.20. The experimental condition of each specimen is given, namely, flux sample number, temperature of heat treatment and holding time respectively. However Figure 3.10 is Metallography image of the sample T-A retrieved specimen from the meniscus

SAMPLE T-A Etchant: - Nil Magnification:-100X



Fig. 3.10: Sample T-A-Retrieve from meniscus region

SAMPLE T-A Etchant: - Nil Magnification:-100X (As cast & stress relieved structure)

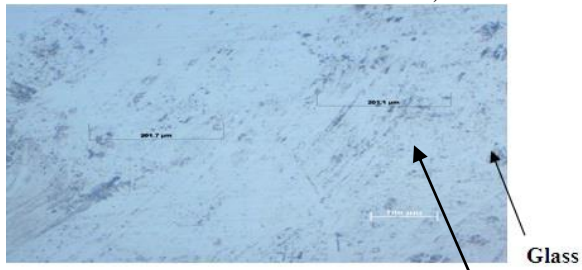


Figure 3.11: T-A As cast and stress Relieved at 600°C

SAMPLE T-A
(Heat treat temperature 800°C - Holding time 2hrs)

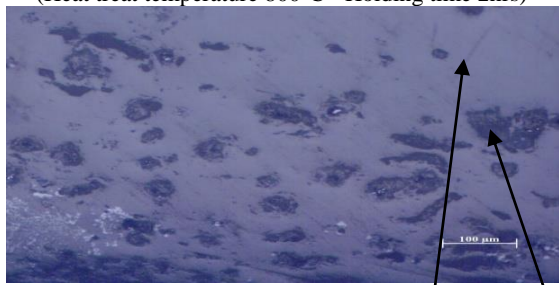


Figure 3.12: T-A - 800°C - 2hrs

SAMPLE T-A
(Heat treat temperature 800°C - Holding time 4hrs)

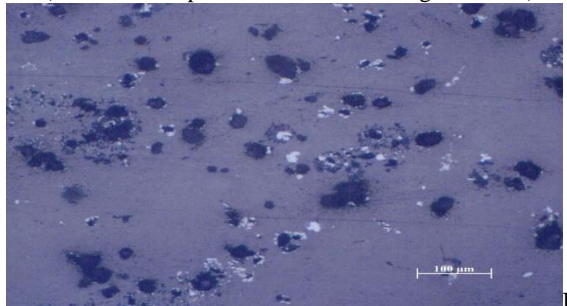


Figure 3.13: T-A - 800°C - 4hrs

SAMPLE T-A
(Heat treat temperature 800°C - Holding time 6hrs)

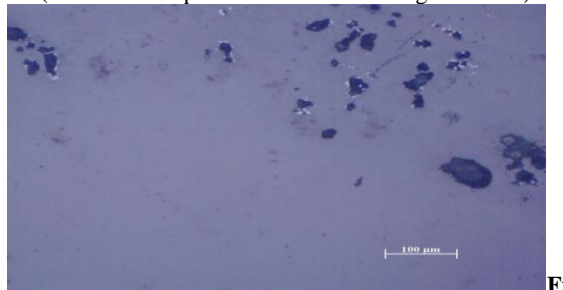


Figure 3.14: T-A - 800°C - 6hrs

SAMPLE T-A
(Heat treat temperature 900°C - Holding time 2hrs)

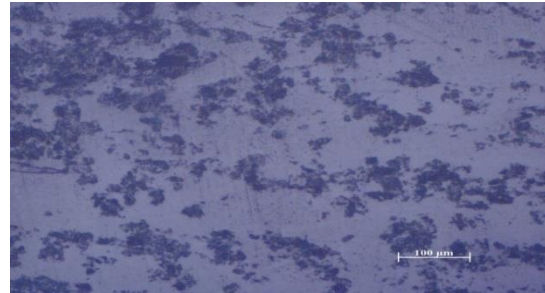


Figure 3.15: T-A - 900°C - 2hrs

SAMPLE T-A
(Heat treat temperature 900°C - Holding time 4hrs)

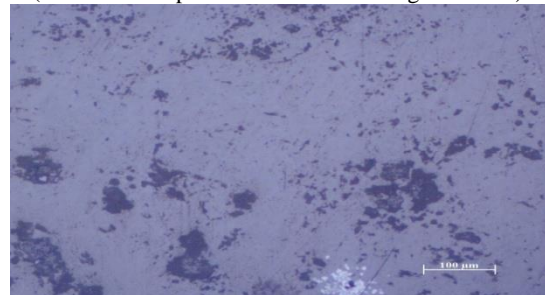


Figure 3.16: T-A - 900°C - 4hrs

SAMPLE T-A
(Heat treat temperature 900°C - Holding time 6hrs)

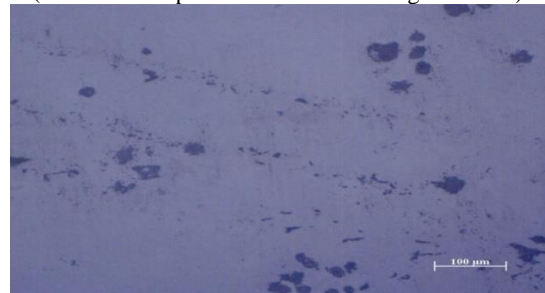


Figure 3.17: T-A - 900°C - 6hrs

SAMPLE T-A
(Heat treat temperature 1000°C - Holding time 2hrs)

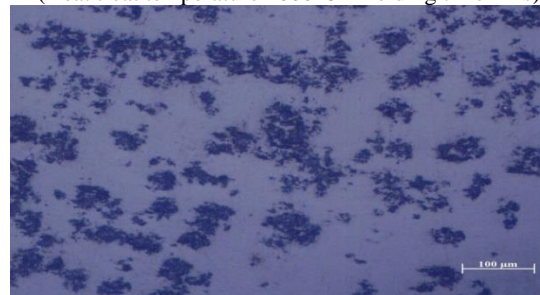


Figure 3.18: T-A - 1000°C - 2hrs

SAMPLE T-A
(Heat treat temperature 1000⁰C - Holding time 4hrs)

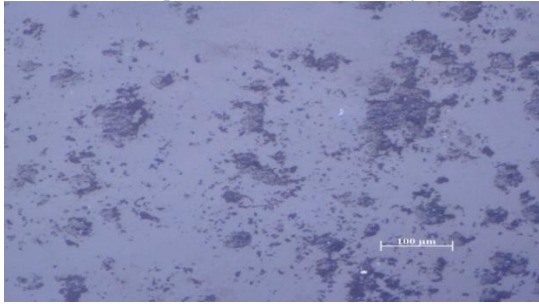


Figure 3.19: T-A - 1000⁰C - 4hrs

SAMPLE T-A
(Heat treat temperature 1000⁰C - Holding time 6hrs)

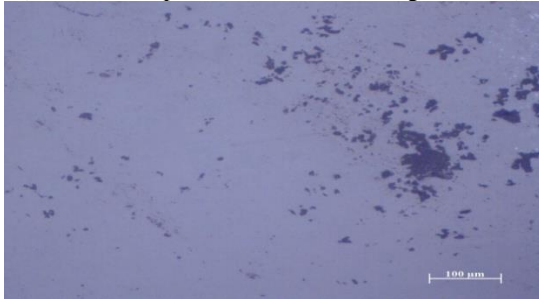


Figure 3.20: T-A - 1000⁰C - 6hrs

Analysis Of Microstructure

Microstructure of each specimen is comprised of bright and dark areas. Bright area went on increasing at the expense of dark areas with increase in heat treating temperature and that with holding time. This was commensurate with the observation made for XRD graphs, in which with increase in temperature of heat treatment and holding time percent cuspidine has found increasing. Thus bright areas in the microstructure are that of crystalline phases and the dark areas are of glassy or amorphous phase.

The crystalline phase area was analyzed with the help of software attached to the Image Analyser in order to obtain fraction of the same under different conditions of experiments.

For this purpose four representative areas were chosen of a given specimen and average percent of crystalline phase of each sample was obtained. This image analysis of all samples under different experimental conditions is summerised in Table 3.2 A, 3.2 B, 3.2 C for flux samples T-1, T-A and T-2 respectively.

Table: 3.2A Percent crystallinity versus heat treating temp. and hold time for sample T-1

Sr. No.	Sample	% Crystallinity
1	T-1 As cast and stress relieved at 600 ⁰ C	14.74
2	T-1 – Heat treat temp. 800 ⁰ C - Holding time 2hrs	60.24
3	T-1 – Heat treat temp. 800 ⁰ C - Holding time 4hrs	64.75
4	T-1 – Heat treat temp. 800 ⁰ C - Holding time 6hrs	65.02
5	T-1 – Heat treat temp. 900 ⁰ C - Holding time 2hrs	72.24
6	T-1 – Heat treat temp. 900 ⁰ C - Holding time 4hrs	76.43
7	T-1 – Heat treat temp. 900 ⁰ C - Holding time 6hrs	79.02
8	T-1 – Heat treat temp. 1000 ⁰ C - Holding time 2hrs	74.07
9	T-1 – Heat treat temp. 1000 ⁰ C - Holding time 4hrs	76.63
10	T-1 – Heat treat temp. 1000 ⁰ C - Holding time 6hrs	82.45

Table: 3.2B Percent crystallinity versus heat treating temp. and hold time for sample T-A

Sr. No.	Sample	% Crystallinity
1	T-A Retrieve from meniscus	12.44
2	T-A As cast and stress relieved at 600 ⁰ C	62.50
3	T-A – Heat treat temp. 800 ⁰ C - Holding time 2hrs	53.71
4	T-A – Heat treat temp. 800 ⁰ C - Holding time 4hrs	61.93
5	T-A – Heat treat temp. 800 ⁰ C - Holding time 6hrs	66.93
6	T-A – Heat treat temp. 900 ⁰ C - Holding time 2hrs	62.77
7	T-A – Heat treat temp. 900 ⁰ C - Holding time 4hrs	65.43
8	T-A – Heat treat temp. 900 ⁰ C - Holding time 6hrs	70.46
9	T-A – Heat treat temp. 1000 ⁰ C – Holding time 2hrs	55.82
10	T-A – Heat treat temp. 1000 ⁰ C – Holding time 4hrs	64.35
11	T-A – Heat treat temp. 1000 ⁰ C – Holding time 6hrs	74.39

Table: 3.2C Percent crystallinity versus heat treating temp. and hold time for sample T-2

Sr. No.	Sample	% Crystallinity
1	T-2 As cast and stress relieved at 600°C	9.56
2	T-2 – Heat treat temp. 700°C - Holding time 2hrs	48.18
3	T-2 – Heat treat temp. 700°C - Holding time 4hrs	54.95
4	T-2 – Heat treat temp. 700°C - Holding time 6hrs	60.61
5	T-2 – Heat treat temp. 800°C - Holding time 2hrs	52.40
6	T-2 – Heat treat temp. 800°C - Holding time 4hrs	58.47
7	T-2 – Heat treat temp. 800°C - Holding time 6hrs	63.68
8	T-2 – Heat treat temp. 900°C - Holding time 2hrs	62.11
9	T-2 – Heat treat temp. 900°C - Holding time 4hrs	64.17
10	T-2 – Heat treat temp. 900°C - Holding time 6hrs	68.11

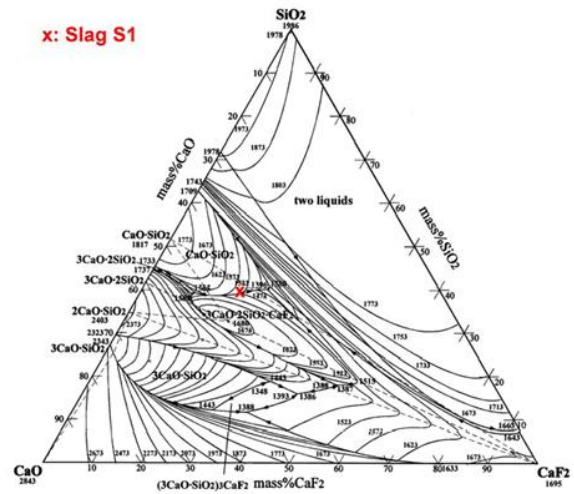


Figure 3.22: Ternary phase diagram CaO–SiO₂ – CaF₂

Findings From X-Ray Diffraction Studies

Some observations made in this respect are given below:

- 1) Variation in percentage area of cuspidine with respect to different temperatures of heat treatment and time period is shown in figure 3.21.
- 2) It was noticed that cuspidine phase is the major constituent of the crystalline phase.
- 3) The performance of T-A sample was found in between the two other fluxes, namely T-1 and T-2. This seems to be the main reason that T-A flux performed well and gave longitudinal crack free casting in actual process carried out in the plant.
- 4) Irrespective of the fact that the castings of all the fluxes, were cast rapidly and quenched, some amount of crystalline phase did form in these specimen in as cast and stress relieved condition.
- 5) Even in a retrieved mold side sample of T-A flux, few peaks of crystalline phase in its XRD graph are seen (figure 3.8) indicating presence of some amount of crystalline phase at that quenched condition.
- 6) Fraction of cuspidine increased rapidly during heat treatment carried out at 800°C compared to that at 900°C and 1000°C.
- 7) Fraction of cuspidine found to increase with time at all temperatures of transformation, except for T-2 fluxes for which fraction of cuspidine decreased marginally at 900°C from 2hrs to 6hrs. this flux has basicity of 0.77. It is possible that cuspidine dissociated into some other compounds such as wollastanite and/or other fluorine containing compounds.

Discussion

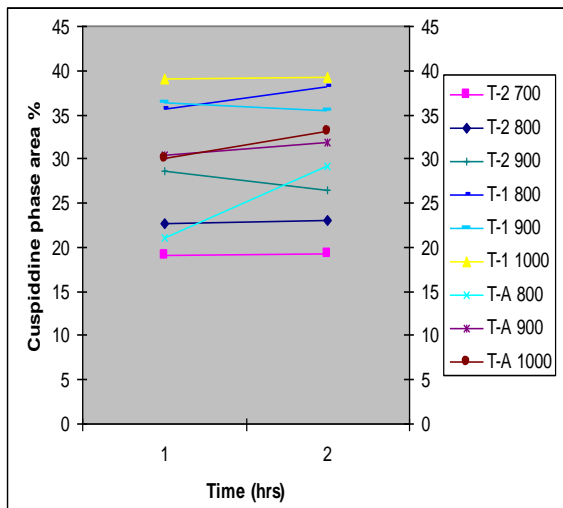


Figure 3.21: Percent of cuspidine as a fraction of temperature and time

Kashiwaya et al [10] has also noted while carrying out high temperature X-Ray Diffraction study, that cuspidine is unstable at high temperatures. It can be seen from the ternary diagram of CaO - SiO₂ - CaF₂, which is shown in figure 3.22 that for the

composition of fluxes under consideration, the other phases which are in equilibrium with cuspidine are $2\text{CaO} \cdot \text{SiO}_2$, $\text{CaO} \cdot \text{SiO}_2$. However this ternary diagram does not take into account effect of Na_2O , K_2O and Li_2O which are also present in concast fluxes.

8) The copper mold side of the retrieved T-A specimen was approximately 100% glassy and the strand side part was fully crystalline (figure 3.9) as it was in contact with steel strand which was approximate at 1400°C relatively a high temperature. Assuming that this sample of T-A stayed in the mold for the entire heat, for one hour duration, it has been found to be fully crystalline. It shows that temperature plays more important role in glassy to crystalline transformation than the time period.

Metallography

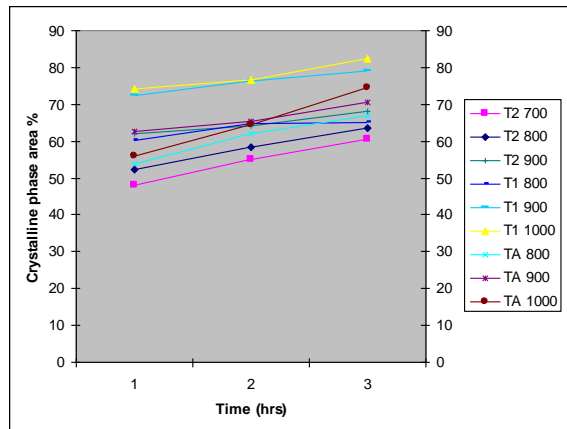


Figure 3.23: Percent of crystalline phase as a function of temperature and time

- 1) Metallographic technique has been established to obtain microstructures of materials like slag, fluxes etc.
- 2) The microstructures obtained in the present work were found similar to one reported in the literature.
- 3) Metallographic technique has given microstructures of all the crystalline components put together as bright area and it was not possible to distinguish between the various constituents within the crystalline phase. For this purpose perhaps techniques employed by geologist would prove more useful.
- 4) Percent area shown by metallographic technique is on the higher side as it can be seen from figure 3.23 compared to percent cuspidine obtained by X-Ray Diffraction graphs. This is obvious as metallographic technique has given information about all crystalline constituents put together which may include cuspidine, wollastanite, nepheline etc.

5) As period of heat treatment increases crystalline fraction was found to increase.

Conclusion

Longitudinal cracking which takes place during continuous casting of peritectic steels is a matter of concern and needs utmost attention. It is possible to contain this problem by controlling horizontal rate of heat transfer through proper selection of mold flux. It is known that by enhancing fraction of crystalline content in the mold flux it is possible to optimize the rate of horizontal heat transfer.

At the plant three mold fluxes were chosen to try in order to avoid longitudinal crack in their castings, selection of which was based on the data available on viscosity and basicity. One of these fluxes worked well. Why this flux performed well was subject of present investigation.

The three fluxes were decarbed, melted and the thin castings of the same were cast and quenched and then subjected it to stress relieving treatment at 600°C for one hour. The samples thus prepared were subjected to isothermal transformation of glassy to crystalline phase at various temperatures and time.

X-Ray Diffraction technique using α -Alumina as external indicator was developed to determine the fraction of crystalline phase such as cuspidine, wollastanite and nepheline. Similarly Metallographic method was also established to determine crystallinity. Following conclusions were drawn from the results thus obtained.

- i) Some amount of cuspidine phase was found even in quenched samples.
- ii) With increase in heat treating temperature and holding time, amount of crystalline content increased.
- iii) The sample of T-A flux was found to have in between fraction of crystalline phase compared to other two, which were having extreme values of viscosity and basicity.
- iv) Strand side specimen of T-A flux was found with complete crystalline phase as it was in contact with the highest temperature 1400°C or so. This shows that temperature plays an important role in the process of crystallizations compared to holding time.
- v) T-1 flux has high basicity and crystallization temperature. This causes formation of thin shell which is unable to sustain stresses.
- vi) On the other hand, T-2 flux has low basicity and also low crystallization temperature, as a

result of which thick steel shell forms. This is responsible for the formation of unsustainable stresses leading to longitudinal crack.

vii) Fluxes which can avoid longitudinal crack in peritectic steels should have moderate amount of crystalline content.

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