In the present work, attempt has been made to investigate the influence of Ti on the castability of Al-killed ultra low carbon (ULC) steel, and to establish the possible reasons for the inferior castability of the Ti-bearing ULC steel vis-à-vis Ti-free grades. The work also attempted to identify suitable countermeasures for prevention of inclusion deposition in the submerged entry nozzles (SEN) during continuous casting of Ti-bearing Al-killed ULC steels. Characteristics of inclusions in the submerged entry nozzle deposit and corresponding liquid steel and slag samples from RH degasser and tundish were investigated. The presence of small quantity of Ti-bearing alumina inclusion was identified to be responsible for the extensive melt freezing inside the SEN deposit and poor castability of Ti-bearing Al-killed ULC steels. The influence of all materials contacting liquid steel subsequent to the RH degassing, on the reoxidation behaviour and castability of liquid steel, was assessed from their chemical composition. The present investigation indicated that while reoxidation is bad for the castability of all Al-killed steels, it becomes worse in the case of Ti-bearing Al-killed steels, primarily due to the formation of Ti-bearing inclusions that promote large-scale melt freezing inside the nozzle deposits.

KEY WORDS: nozzle clogging; continuous casting; submerged entry nozzle; ultra low carbon steel; castability; titanium oxide inclusion.
clusters. Further, the calculations by Rackers and Thomas have demonstrated that freezing of metal inside the inclusion deposit reinforces the clog; just as steel bars add strength to the reinforced concrete. The variety of information available in literature point towards the general understanding that degradation of castability of Al-killed low carbon steels in presence of dissolved Ti is caused primarily due to reoxidation of liquid steel and formation of inclusions that easily deposit at the nozzle refractory surface.

Production of Ti-bearing ULC interstitial free (IF) steel at Tata Steel had been suffering from high rejection of the cold rolled sheets, mainly due to the presence of slivers. The inferior castability of Ti-bearing steel in comparison with that of Ti-free low carbon steels was identified as one of the main reasons of slivers. Therefore, the present work was undertaken to identify the reasons for the deterioration of castability of Ti-bearing Al-killed ultra low carbon steel and attempts have been made to recommend suitable countermeasures to minimise the occurrence of nozzle clogging.

2. Experimental

The methodology adopted in the present investigation included:
(a) collection of inclusion deposit from the submerged entry nozzle (SEN) and corresponding liquid steel and slag samples from ladle and tundish,
(b) characterization of inclusions in liquid steel samples using SEM-EDX,
(c) identification of the sources of clogging agents.

Figure 2 presents schematic of the processing route of Al-killed ULC steels at Tata Steel. The typical chemical compositions of the Ti-bearing and Ti-free steel grades are shown in Table 1. A total of 40 SENs and corresponding samples of liquid steel and slag of Ti-bearing, as well as Ti-free, ULC grades were examined during the present work. Physical Characteristics of SEN deposits were visually examined. For identification of various clogging agents in the liquid steel, samples were collected from different layers of clog and examined in the scanning electron microscope (SEM) and energy dispersive spectroscopy (EDX). Subsequently, the origins of different clogging agents were traced back through the SEM-EDX examination of inclusions in the corresponding liquid steel samples from the ladle metallurgy stage and the tundish. Chemical compositions of ladle and tundish slags of corresponding heats were also examined in order to assess their role in reoxidation of liquid steel and generation of secondary inclusions. In addition, compositions of all materials contacting liquid steel subsequent to the ladle treatment were also examined to estimate their influence on nozzle clogging.

3. Results and Discussions

3.1. Visual Observations

Majority of the deposits were observed at the SEN bottom, typically in the form of a conical pile of 80–100 mm height. This was observed systematically in all SENs, though the quantity and dimension varied. The extent of deposition was much less inside the barrel of the SEN (Fig. 3(a)) compared to the port area (Fig. 3(b)). The total

|            | C    | Mn  | Si  | S   | P   | Al  | Nb  | Ti  | N   |
|------------|------|-----|-----|-----|-----|-----|-----|-----|-----|
| Ti-bearing | 0.0025 | 0.12 | 0.008 | 0.007 | 0.012 | 0.045 | 0.012 | 0.050 | 0.0030 |
| Ti-free    | 0.020 | 0.18 | 0.008 | 0.007 | 0.012 | 0.045 | –   | –   | 0.0035 |
volume of deposits observed in the case of Ti-free grades was relatively less than in the case of Ti-bearing grades. The inclusion deposits appeared whitish (indicated by the white arrow in Fig. 3(b)) and were radially symmetric inside the SEN, irrespective of the steel grade. Figure 3(c) shows a typical clog sample removed from the base of a SEN that was used for casting 700 tonnes of Ti-bearing ULC steel, covering a total casting time of 335 min. The broken line indicates the position of the base of the SEN and the exist ports, which have been removed to expose the deposit inside. The major macroscopic features of the clog samples, collected from inside the SEN, are presented in Table 2.

### 3.2. Characteristics of Inclusions in SEN Deposits

Table 2 presents the macroscopic observations of deposits collected from different layers of the SEN.

| Item                        | Deposits in Ti-Al steel | Deposits in Ti-free steel |
|-----------------------------|-------------------------|---------------------------|
| Volume of deposits          | more in volume          | less in volume            |
| Shape                       | conical                 | conical                   |
| Density                     | heavier                 | lighter                   |
| Colour                      | greyish white           | chalky white              |
| Friability                  | tough                   | more friable              |
| Presence of entrapped steel | large amount of frozen steel throughout the deposit | occasional localized freezing of globular steel particles (more towards metal side & occasionally towards refractory side) |
| Principal clog constituent | coarse Al₂O₃ & spinel inclusions | coral shaped clusters of pure Al₂O₃ |
| Presence of spinel inclusions | upto approx. 15-20 vol.% spinel present along with alumina clusters in nozzle deposits | small quantity of spinel found at a few places in nozzle deposits |
| Presence of Ti-bearing inclusions | fine Ti-bearing inclusions (TiO₂-Al₂O₃) observed at few locations | no trace of Ti bearing inclusions |
| Al₂O₃ inclusion size        | mixed size distribution of alumina grains | individual alumina particle relatively finer |

* after similar casting duration

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Fig. 3. (a) Negligible deposition in the barrel of a SEN (after 6 h of use). (b) Transverse section of the SEN, cut at about 10 cm above the port. (c) Bulk and typical shape of the deposit removed from the port of the same SEN.
posits were characterised using SEM-EDX. The nature of clog was found to be highly heterogeneous in all the samples studied. However, the bulk of the deposits consisted of coral shaped clusters of pure alumina (Al$_2$O$_3$). Minor constituents included spinel (MgO–Al$_2$O$_3$) inclusions and occasional presence of spherical droplets of frozen steel. The area percent of spinel in the inclusion deposits determined from the image analyser was found to vary from 5 to 10%. In addition to these constituents, the deposits in the case of Ti-bearing ULC steel were observed to contain Ti-bearing alumina (TiO$_x$–Al$_2$O$_3$) inclusions and large amounts of frozen steel throughout the deposits. A typical SEM micrograph of alumina cluster along with its X-ray elemental map is shown in Figs. 4 and 5. It can be seen clearly from Fig. 5 that entrapment of steel in the nozzle deposits occurred in immediate vicinity of TiO$_x$–Al$_2$O$_3$ deposits, in the case of Ti-bearing steel grades.

It has been concluded that the reason for the difference in the shape and amount of entrapped steel frozen inside the inclusion clusters lies in the variation in the wettability of the different inclusions towards liquid steel. As reported in literature, TiO$_x$–Al$_2$O$_3$ inclusions are easily wetted by liquid steel. Liquid steel thus penetrates through the pores in the clusters containing TiO$_x$–Al$_2$O$_3$, and freezes in the form of patches. This may explain the preferential freezing of steel in immediate vicinity of the Ti-bearing alumina inclusion clusters, formed during the casting of Ti-bearing Al-killed ULC steel (e.g. IF steel).

Samples of liquid steel and slag were collected from the tundish and the ladle to trace back the origin of various clogging agents observed in the nozzle deposits of Ti-bearing and Ti-free steel grades. Isolated angular alumina particles (size <5 μm) were the most common constituents, along with a few compact alumina clusters in some cases. The area fraction of spinel in the nozzle deposits went up to 20% in the case of Ti-bearing grades. However, the presence of Ti was found to cause no major difference in the gross morphological features of alumina clusters and this appeared to be in variance with the observations of some earlier investigators.

3.3. Role of Deoxidation in Formation of Clog

It was difficult to estimate the exact stoichiometry of association of Ti with the alumina inclusions. However, observation of the morphology of the inclusions and thermodynamic analysis of the same helped to throw some light on this issue. Ti associated with alumina could be present either as TiN$_x$ or TiO$_x$. Skoczylas and co-workers have reported that at least 68 ppm dissolved [N] were required for precipitation of TiN inclusions at 1525°C if the liquid steel contained 0.23 mass% dissolved [Ti]. The Ti-bearing ULC steel at Tata Steel contains approximately 0.05 mass% [Ti]. Calculations using standard thermodynamic data and equations similar to those reported by Skoczylas et al. has revealed that at least 350 ppm [N] concentration would be necessary to initiate TiN precipitation at 1550°C ($x=1$). The concentration of dissolved nitrogen in all the samples of Ti-bearing ULC steel were below 30 ppm and the possibility of TiN$_x$ precipitation in the liquid steel was
therefore ruled out. In addition, TiN, inclusion typically has a cuboidal shape, which was not observed in any of the clog samples investigated. It was thus concluded that TiO, associated with Al₂O₃, was the most likely form of titanium found in the inclusions observed in Ti-bearing ULC steel samples. Based on the understanding developed from the works of Lehman and Gaye, it could be concluded that re-oxidation of liquid Ti-bearing ULC steel in the ladle and/or the tundish during casting was the most likely source of TiOₓ–Al₂O₃ inclusions.

Figures 6 and 7 show the scanning electron micrographs, along with the elemental map, of some of the inclusions observed in the samples of Ti-bearing ULC steel, collected from the tundish. It can be seen that the surfaces of pure alumina clusters are partially (or completely) covered with TiOₓ–Al₂O₃ inclusions. Pre-existing solid alumina inclusions seemed to act as preferred sites for deposition of TiOₓ–Al₂O₃ inclusions, partially resembling the morphology of oxide–sulphide duplex inclusions. The presence of TiOₓ–Al₂O₃ inclusions was a unique feature of Ti-bearing steel, not observed in any of the Ti-free steel samples. It is evident from the inclusion precipitation diagram in Fig. 1 that Al₂O₃ would indeed be the primary deoxidation product, even in the presence of Ti, for the range of chemistry under study. TiO may form only if oxygen is subsequently made available in the system, i.e. through reoxidation. Further, the extent of fading of dissolved Al and Ti between the samples collected from RH degasser and from the tundish, illustrated in Fig. 8, clearly indicated towards reoxidation of liquid steel. Average fading of 100–110 ppm of [Al] and ~70 ppm of [Ti] was observed over a time interval of 40–50 min.

To prevent re-oxidation, liquid Al-killed steel must be well insulated from any contact with air at all stages subsequent to secondary metallurgy treatment. In addition, all materials contacting liquid steel must be practically free from reducible oxides like FeO, MnO and SiO₂. Attempt has been made to identify the potential sources of re-oxidation during the processing of Ti-bearing ULC steel, and devise means to minimise their detrimental effect.

3.4. Identification of the Sources of Reoxidation

The extent of [N] pick-up less than 2 ppm (by mass) or less in all the samples studied, which indicated that aerial reoxidation played only a minor role and contact with air was effectively avoided. Therefore, the search for potential sources of reoxidation was restricted to the slag layers in ladle and tundish, and the refractory materials coming in contact with liquid steel after the RH degassing operation.

(a) Ladle Slag

Chemical analysis of the ladle slag, collected at the end of ladle metallurgy stage, revealed an average chemistry of 13–16 mass% Fe (in form of FeOₓ), 38–48 mass% CaO, 10–18 mass% Al₂O₃ and 5–6 mass% SiO₂, along with small quantities of MnO and MgO. The total reducible oxide concentration (FeOₓ+MnO) was always in excess of 15 mass%. Slag FeO in excess of 12% has been reported[17] to be oxi-
dising to the dissolved Al and Ti in liquid steel and this was therefore identified as a potential source of reoxidation in the ladle.

Consequent to this finding, conditioning of the ladle slag by addition of ferro–aluminium (Fe–Al) chips was adopted. The quantity of addition, starting at 50 kg per heat, has been gradually increased and is now stabilized at 150 kg of Fe–Al chips per heat. Figure 9 shows the progressive reduction in FeO and MnO content in the ladle slag since adoption of this practice.

(b) Tundish Slag

A combination of two different powders was used in the tundish during casting of all ultralow carbon heats at Tata Steel. The dual flux system was intended to simultaneously provide thermal insulation, inclusion absorption and protection against reoxidation of liquid steel.18) The specification of basic tundish covering powder commonly used during casting of Ti-bearing ULC grade of steel was 46–50 mass% CaO, 42–45 mass% Al2O3, 3 mass% (max) SiO2, 2 mass% (max) Fe2O3 and 1.8–2.5 mass% TiO2, and the liquidus temperature was around 1 380°C. Steady increase of SiO2 concentration in the tundish slag was observed in all the casting sequences studied. The general trend of increase in the silica content of slag is shown in Fig. 10. Visual observations during the casting process revealed that the basic tundish-covering powder melted quickly upon addition to the tundish. As a result, the layer of molten basic flux came in direct contact with the layer of burnt rice husk having 94 mass% SiO2, causing dissolution of SiO2 in the molten basic slag layer. The resulting rise in SiO2 content caused the tundish slag to lose its basicity, and it became oxidising to the liquid steel.

High silica content of tundish slag is oxidising to Al and Ti dissolved in liquid steel and its contact with Ti-bearing Al-killed low carbon steel causes generation of secondary inclusions.

\[
\frac{6+x}{2} (\text{SiO}_2) + 4\text{Al} + \text{Ti} \rightarrow \frac{6+x}{2} \text{Si} + (\text{Al}_2\text{O}_3)_{\text{precipitate}} + (\text{TiO}_2 - \text{Al}_2\text{O}_3)_{\text{precipitate}} \ldots \ldots (1)
\]

Figure 11 offers further evidence in the form of Si pick-up by liquid steel in the tundish. The average increase in Si content of liquid steel was observed to be in the range of 0.002–0.004 mass%.

Several studies have established that the time taken for complete melting of basic flux should approximately coincide with the total duration of the casting sequence.18) This would prevent dissolution of silica from the top siliceous layer in the tundish since the rice husk would remain in contact with solid basic flux almost till the end of the casting sequence. Accordingly, it was concluded that reoxidation of liquid steel due to silica dissolution in the tundish slag could be partially reduced by increasing the thickness of the basic covering powder in the tundish.

Trials were conducted by increasing the quantity of basic tundish-covering powder from 50 to 100 kg during casting of Ti-bearing Al-killed ultra low carbon steels. This led to further reduction in the nozzle clogging during slab casting, measured in terms of mould level fluctuations and the number of flushes per heat.

(c) Ladle Nozzle-well Packing Material

Tanaka and others17) have suggested that the ladle nozzle packing material should be based on Al2O3 or Cr2O3, with the total reducible oxide content (SiO2 + FeO + MnO) less than 10 mass%. This would ensure chemical inertness of the powder towards liquid steel. The ladle nozzle packing powder, used with all the ULC heats, contained 50–60 mass% SiO2, 20–26 mass% Cr2O3, 5–7 mass% Fe2O3, 4–6 mass% MgO, 3–5 mass% Al2O3 and trace quantities of CaO.
and TiO₂. Since the total reducible content was clearly in excess of 50 mass\%, it could be inferred that the SiO₂-rich nozzle packing material was also a likely source for reoxidation of liquid ULC steel.

(d) Alumina Graphite SEN Refractory

After completion of every casting sequence, the inner surface of the submerged entry nozzle was found to contain a dense layer of nearly-pure Al₂O₃ precipitates along with surface quantities of alkali oxides. The alumina-graphite refractory contained 64 mass\% Al₂O₃, 6 mass\% SiO₂, 22 mass\% C, 4.6 mass\% ZrO₂, 0.4 mass\% Fe₂O₃, 1.8 mass\% B₂O₃ and traces of alkali oxides. SiO₂ and traces of alkali are common impurities in the alumina-graphite refractory of commercial submerged entry nozzles. With rise in temperature during preheating and after start of casting, these impurities undergo carbothermic reduction with the graphite of the refractory and release gaseous SiO and CO. These gases are oxidising to aluminium dissolved in liquid steel and cause in-situ precipitation of alumina on the SEN surface. The following reactions are believed to be responsible for this.21)

\[
\text{SiO}_2(s) + 2\text{C}(s) \rightarrow \text{SiO}(g) + \text{CO}(g) \quad \text{(2)}
\]

\[
\text{Na}_2\text{O}(s) + \text{C}(s) \rightarrow 2\text{Na} + \text{CO}(g) \quad \text{(3)}
\]

\[
3\text{CO}(g) + 2\text{Al} \rightarrow 3\text{C} + (\text{Al}_2\text{O}_3)_{\text{precipitate}} \quad \text{(4)}
\]

\[
3\text{SiO}(g) + 2\text{Al} \rightarrow 3\text{Si} + (\text{Al}_2\text{O}_3)_{\text{precipitate}} \quad \text{(5)}
\]

In-situ precipitation of alumina occurs almost instantaneously once the clean SEN comes in direct contact with the liquid steel at the beginning of the casting sequence.20) Thereafter, the already deposited inclusions act as nuclei for further deposition at the nozzle wall. This has been reported as a serious limitation of the use of alumina-graphite nozzles in continuous casting of Al-killed ULC steel.20,22,24) To overcome this problem, some researchers have attempted to develop casting nozzles with internal lining of zirconia, boron nitride or calcia-zirconia, or develop carbon-free nozzle refractory. It may therefore be inferred that controlling the initial precipitation of alumina on the SEN refractory surface is the most appropriate means to minimize clogging of the SEN. This may be achieved by minimising the silica and alkali impurities in alumina-graphite refractory. Trials elsewhere with 72.4 mass\% Al₂O₃ and 1.0 mass\% SiO₂ in the refractory material have reported significant improvements. In view of this, the plant was advised to use low-impurity (less than 3 mass\% reducible oxides) SENs for casting of Ti-bearing ultra low carbon steels.

(e) Tundish Working Lining

The conventional MgO based tundish working lining, containing 65–72 mass\% MgO and 20–25 mass\% SiO₂, was also a potent source of re-oxidation.25,26) The total reducible oxide (SiO₂ + FeO + MnO) content in the tundish lining (sprayable) material should be less than 10 mass\% for an optimum performance.17) This would ensure inertness of the refractory material to liquid Al-killed steel and would help in reducing the problem of secondary inclusion (clogging agent) formation due to re-oxidation.

Some steel plants have adopted the practice of six-surface reactive contact, where the tundish internal surfaces are made reactive with a calcite (CaCO₃) based working lining.27) In that case, all six surfaces of the tundish participate in the inclusion removal and ensure a higher cleanliness level of the liquid steel before its delivery to the mould. The unfloated alumina inclusions have a higher probability of getting absorbed by the CaO present in the tundish lining, than in the case of conventional MgO–SiO₂ based lining. Success of this technique, however, depends strongly upon proper firing of the calcite lining prior to start of cast. Lime coating on the tundish internal surface was also tried at Tata Steel as a cheaper alternative, but it did not ensure consistent alumina removal owing to the short life of the coating.19)

4. Conclusions

The difference in castability behaviour of Ti-free and Ti-bearing ultra low carbon steels has been investigated based on the characteristics of the inclusions found in the SEN deposits and in the corresponding liquid steel samples of these grades of steel. The salient findings of the present investigation were as follows:

(1) Inclusion deposits found in the case of Ti-free ULC grade were friable (chalky), containing mostly coral shaped alumina clusters and spinel (Al₂O₃–MgO) inclusions, along with a few frozen steel droplets inside the deposits. In contrast to this, the deposits formed with Ti-bearing steel contained large quantities of frozen steel dispersed throughout the nozzle deposit in the form of layers and patches, along with the clusters of alumina and spinel inclusions.

(2) Extensive melt freezing was observed throughout the deposits in the case of Ti-bearing ULC steel. Ti-bearing alumina (TiO₂–Al₂O₃) inclusions were observed in the alumina clusters immediately adjacent to frozen steel. Based on thermodynamic analysis and observations of other researchers, entrapment of steel inside the inclusion clusters was attributed to the wetting characteristics of TiO₂–Al₂O₃ inclusions.

(3) Deposition of inclusion clusters inside the SEN occurred in all Al-killed low carbon and ultra low carbon steels. However, the severity of deposition was much higher in the case of Ti-bearing steel grades, primarily due to the contamination of un-floated alumina inclusions by TiO₂–Al₂O₃, and entrapment of steel inside the TiO₂–Al₂O₃ containing clusters.

(4) Based on thermodynamic considerations, it was concluded that the TiO₂–Al₂O₃ inclusions originated almost entirely due to reoxidation of molten steel during casting. Probable sources of reoxidation were identified and appropriate counter-measures for control of reoxidation were recommended to the operating personnel of the steel melting shop. Adoption of ladle slag conditioning, modification in tundish covering practice and use of high-chromia ladle nozzle packing powder (described in Secs. 3.4(a), (b) and (c) respectively) showed beneficial effect in terms of reduction in the extent of clogging. Subsequently to adoption of these recommendations, the frequency of occurrence reoxidation-related slivers in cold rolled Ti-bearing Al-killed ULC steel sheets was reduced from the earlier level of 80–85% to less than 60%. However, the contributions of the
individual initiatives were difficult to ascertain since the different modifications were implemented simultaneously in the plant.

Acknowledgements

The authors gratefully acknowledge the Management of Tata Steel for granting permission to publish the work. Prof. Ahindra Ghosh is gratefully acknowledged for his valuable suggestions during the work. Authors are also thankful to the In-charge and operating personnel of the LD-2 and the Slab Caster Shops for their co-operation in sample and data collection. The authors wish to extend special thanks to Mr. Vikram Sharma and Mr. A. J. Khan for carrying out extensive SEM-EDX work.

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