By use of a multi-depth sampling technique at key steps during the ladle metallurgy furnace process, the evolution of inclusions can be observed up to depths of 1.85 m below the slag layer in both Si-bearing and restricted Si (RSi) grades. This combination of data showed the effects of silicon on the slag/steel interaction. The Si-bearing aluminum-killed grades had different inclusion distributions, populations, as well as morphologies than the RSi grades. It was also seen that there is a sulfur wt.% threshold that affects the ability for the slag to interact with the inclusions in the steel. In return, the inclusion morphology for RSi heats can mimic that of silicon-bearing heats with low final sulfur wt.%. This threshold was determined to be 0.003 wt.% sulfur in the steel.

During the ladle metallurgy furnace (LMF) process, there are several key processes that must occur in order to produce a heat suitable for customers’ requirements. For basic aluminum-killed heats, deoxidation (Eq. 1), desulfurization (Eq. 2), slag formation, inclusion removal, temperature control and calcium treatment are among the most critical parts of the operation. Each of these components is of interest to many researchers.

Inclusion formation is inevitable. Alumina is present in steel as an inclusion from either deoxidation or reoxidation. It is an inclusion to be studied since it can be detrimental to the castability and cleanliness of the final product. Throughout the processing at the LMF, alumina inclusions can interact with the refractories along with some residual MgO in alloys to form spinels. The inclusion evolution throughout a heat has been studied by several researchers. These studies all have shown that inclusions tend to follow a very similar inclusion path from alumina (Al₂O₃) to spinel to Ca-Al or a liquid phase. An example of the inclusion evolution is shown in Fig. 1. However, there are several processing variables that can affect the path of the inclusions, increase their size and/or change the population of the inclusions.

\[
2[\text{Al}] + 3[O] \rightarrow (\text{Al}_2\text{O}_3) \tag{Eq. 1}
\]

\[
3(\text{CaO}) + 2[\text{Al}] + 3[\text{S}] \rightarrow (\text{Al}_2\text{O}_3) + 3(\text{CaS}) \tag{Eq. 2}
\]

The addition of materials can have a direct impact on the quality of the steel. One particular element that can have a major effect on the steel is silicon. FeSi is a major source to add silicon to the heat. However, due to the residual Ca wt.% in FeSi, it has been seen that the timing of the addition can affect inclusion concentration, distribution and modification. When FeSi is added early in the heat, the residual calcium has been seen to aid in desulfurization. Silicon can also be used...
as a deoxidizer, though due to its inability to reduce oxygen level as low as that of aluminum, it is not used in applications where oxygen concentration is a concern. For Al-killed steel, silicon is added to increase the physical properties. However, Si-bearing heats react differently than those with a silicon restriction (RSi). RSi heats are typically restricted to a 0.04 wt.% Si maximum. Si-bearing heats typically have less aluminum consumption than RSi heats, and tend to desulfurize more efficiently. The silicon present in the heat also changes slag properties and inclusion composition distribution. This typically results in a cleaner heat at the LMF.

Experimental Procedure

A multi-depth sampling technique was created, which allowed for simultaneous sampling at various depths of 6 inches (0.15 m), 30 inches (0.76 m) and 54 inches (1.37 m) beneath the slag layer. The validation of the tube, as well as the construction of the tube, was presented previously in Reference 13. A schematic of the tube is shown in Fig. 2. This sample set was taken in conjunction with Nucor Steel Tuscaloosa Inc.’s (NSTI) standard robot sample along with temperature and slag samples throughout key process steps. The key process steps are shown in Fig. 3, and include heat arrival/starting (S), after alloying (A), after desulfurization/the start of pre-rinse (R), prior to calcium
treatment/end of pre-rinse (C) and after the end of post-rinse/end of heat (E). The sample time nomenclature will be used in conjunction with the sampling depth where (1) is the sample closest to the slag layer, (2) is approximately 30 inches down and (3) is the deepest sample.

Since the back side of the ladle at NSTI is not accessible, the samples were taken from one porous plug location. It is assumed the flow in the ladle is symmetric due to the plug locations. Fig. 4 illustrates the layout of the hood design. It also includes the locations for sampling and alloying. Four steel samples were taken at the same sample time. This included the multi-depth sampler, which was located on the downward side of the stir; a robot sample, which is located on the side of the stir path; and a temperature taken with the robot arm as well. The robot sample depth is relatively close to the same depth as sample 2 in the multi-depth tube. The chemistries for the lollipop samples and the multi-depth samples were analyzed using a spectrometer. These samples were also used to complete automated feature analysis (AFA) in a scanning electron microscope (SEM). The AFA produces numerous outputs, one of which is called the inclusion index. The inclusion index is a ratio of the total summed inclusion surface area to the area that was scanned by the AFA program (~160 mm²). The index allows for a baseline comparison for AFAs. The index is one of several components used as a factor in rating steel cleanliness at NSTI. The results from these tests were compiled and compared.

Results and Discussion

One of the main reasons the multi-depth sampling tube was created was to be able to look at inclusion evolution not only through the LMF process but also at multiple depths simultaneously. The use of this tool grants the ability to see inclusion evolution, distribution, morphology and compositional changes throughout the process. AFA analysis was conducted on all the steel samples collected. Fig. 5 shows the multi-depth samples from heat RSI3. This figure shows the R, C and E processing steps at the different depths 1, 2 and 3. The figure shows very little change in the inclusion average location (represented as a blue square) as the sample set gets deeper; however,
there is a slight increase in tail formation toward the modification region/liquid region (blue highlighted region) as the sample depth increases. The inclusion count reduces as the sample depth increases in both the R and C samples. This implies that the slag layer is the most reactive layer during processing until Ca treatment has taken place as shown in sample E. The sample E set shows that the modification region is shifted slightly more to the Ca corner of the liquid region.

Fig. 6 shows the robot samples taken in conjunction with the multi-depth samples. The robot samples at times R and C show a higher inclusion index, count and much longer tail than the samples taken from the same time using the multi-depth. With the robot sample located on the side of the stir path, this observation seems unusual. However, after Ca treatment (sample E), the inclusion index on the robot sample was seen to be much less than those of the multi-depth samples. For this RSi heat, the robot samples vary significantly from the multi-depth samples with the exception of the final sample E.

In view of SI3 in Fig. 7, a Si-bearing heat, it can be noticed that the same trends that were present for RSi3 are not present for SI3. The inclusion index, population, distribution and counts do not significantly vary. All the Si-bearing heats in this sample set were in agreement. The tail formation slightly increases at the deeper depths, but not as quantifiable as the RSi heats. It can be noticed that the %Ca does not always increase as the depth increases. On the final sample E, the deepest sample is the most modified, and has the lowest number of inclusions in comparison to the higher depths. When comparing the multi-depth samples to the robot samples for Si-bearing heats, the differences are vividly dramatic, as shown
Looking into the differences between the RSi heats versus Si-bearing heats, some of the AFA results were plotted side by side. Fig. 9 shows the inclusions index versus all the processing steps and samples for heats RSi3 and SI3. As stated previously, the variation from the robot samples is seen throughout both heats and all sample sets. The figure also shows that the arrival sample S has a significant amount of variability on both heats. This is explained by the amount of turbulence during the early stages in the heat due to high stir rates. The Si-bearing heat generally showed less variability throughout the sample set, and all samples in the Si-bearing heat finished with a significantly lower inclusion index that the RSi heat.

The Ca/Al ratio is another variable that is typically considered since targeting a liquid region for inclusions is desired. Breaking down the components of the Ca/Al ratio into %Al and %Ca, Fig. 10 and Fig. 11, respectively, compare the differences of RSi and Si-bearing heats. Neither chart set was unexpected. Since it has been seen that Si-bearing heats tend to have less aluminum consumption, the SI3 heat showed less %Al on all sample sets except sample S. The charts for %Ca showed that the Si-bearing heats tend to show more interaction throughout the heat relating to an increase in %Ca in the inclusion composition. A better explanation of this will be provided.

Fig. 12 shows the % solid inclusions during the LMF process steps. There is a significant difference in the trend between Si-bearing and RSi. Though the RSi3 heat does show some reduction of % solid throughout the heat, SI3 shows a significant change throughout each processing step. The final step was not plotted due to it being more than 95% liquid due to Ca treatment. These charts are similar to what is depicted in Fig. 11.

While comparing the heats in this study, a significant difference was seen in the inclusion counts from samples C to E. This is
illustrated in Fig. 13, which shows that the inclusion counts in the sample before Ca treatment (blue) are always lower than the final sample inclusion counts. This chart also shows the inclusion counts for Si-bearing grades have less inclusions present than RSi in most cases. It was also noted that a higher inclusion count does not always indicate a higher inclusion index. As seen in Fig. 14, the inclusion index for both RSi and Si-bearing heats decline after wire.

It has been shown that inclusion distributions are affected by different means. In an attempt to understand the variability that has been shown, the inclusion paths were plotted on the same chart (Fig. 15). This figure shows the inclusion evolution plots broken down by RSi and Si-bearing heats. The AFA average point for each process step was plotted and connected to form the lines shown. This figure also includes the pre-wire, sample C, sulfur and silicon for each heat. After the lines were plotted together, it was noticed that the RSi grades’ inclusion paths were very dependent on the sulfur wt.% in the steel. RSI2 and RSI3 heat plots were identical. As the sulfur wt.% increases in the steel, the peak of the inclusion path becomes lower on the chart. The final sample point has a very similar location on both sets of graphs. However, it was noted that the final sample point for Si-bearing grades is closer to the Ca corner of the ternary. For the Si-bearing heats, the lines overlap very well on all but one heat. This particular heat had excessive furnace slag carryover and a higher silicon requirement.

The amount of MgO seen in the inclusions or the height of the inclusion path can be an indicator of oxygen in the steel. The higher the oxygen potential in the system, the slower the desulfurization reaction. Removing the oxygen in the steel not only drives sulfur removal, but it also promotes the increase in spinel formation. As the heat progresses, %MgO increases due to the interaction with the slag as well as the refractories on the ladle walls. After Ca treatment, the %MgO declines. This is due to the Ca modifying the spinel inclusions, causing the %MgO to be reduced as well as not being exposed as frequently to the AFA scan. The plots in Fig. 15 are very similar on all heats except for RSI4. This particular heat had the least amount of processing time (38 minutes) and a high incoming sulfur wt.%.

After the observations were made from the inclusion evolution plots about sulfur content affecting the inclusion path, the attention was directed toward the various AFAs for these particular heats. Typically for
RSi heats, the inclusion compositional grouping is known to be very tight or clustered together (Fig. 16). The compositional grouping on the C sample shows a slight spread; however, the grouping only starts to change after the sulfur reaches below 0.003%. This heat was only below 0.005% for approximately 6 minutes, allowing the grouping to remain fairly tight. However, when looking at another RSi heat in Fig. 17, the grouping starts to spread out, starting in the R sample, significantly sooner than the previous heat. This allowed for more than 18 minutes of processing time before the Ca wire addition, where the sulfur content was low. This resulted in a much larger tail formation toward the liquid area. This heat was slightly depleted of Al between the A and R sampling times, which resulted in a slight increase in sulfur between the two samples.

For Si-bearing heats, the grouping is rarely tight. The tail formation length has been shown to be dependent on a couple of variables. This was initially believed to be related to the Ca present in FeSi. The residual Ca present in FeSi can have an effect on the inclusions especially in a late trim addition after desulfurization has plateaued. This can be seen in Fig. 18, where a 34 kg addition of FeSi was made after the sulfur wt.% was lower, but not below the 0.003 wt.% threshold. This resulted in a slightly extended tail formation. However, it is also seen that sulfur can play a role in the groupings even for Si-bearing heats. This is much more challenging to uncover since typically Si-bearing heats have a tendency to achieve low levels of sulfur easier and more rapidly than RSi heats. An example of a typical Si-bearing AFA with a low sulfur wt.% is shown in Fig. 19. Even though there was a 100 kg (220 lb.) FeSi addition after sample A, the tail formation of the inclusions is much longer in comparison to SI4, which did not desulfurize as low and as quickly as SI3.

Since sulfur is a main driver of the tail formation in AFAs, it also plays a role in the final inclusion distribution. If the initial compositional range is extended, the final grouping after modification...
will also be extended. The tail formation does not shrink in length; however, under circumstances already discussed, it can extend further. This can make it difficult to predict the ideal Ca wire amount needed for inclusion modification. If additional Ca wire is added to the heat in hopes of shifting the inclusion group, it will only form more CaS, not tighten the grouping. This is due to the distribution of Ca on the inclusion population remaining. An example of this is shown in Fig. 20.

Conclusions

The multi-depth sampler allowed for the opportunity to see inclusions at multiple depths simultaneously. This data set was used to explore inclusion distributions, paths, compositional changes, etc. The differences observed between the robot samples and the multi-depth samples were not expected; however, it is believed to be related to the location in the stir pattern, stir velocities and slag properties. It was seen that the Si-bearing heats had minimal change between the various sampling times and locations with respect to inclusion distributions, index and counts. For the RSi heats, there was a considerable change in inclusion population and compositional distribution. Another conclusion was related to the Si-bearing heats, which typically had a better inclusion index/cleanliness and lower inclusions counts on all sampling times and locations. A better understanding of the relationship between slag properties, stir velocities and location is desired, and the work is ongoing.

One of the significant contributions of this study is the effect of sulfur and silicon on inclusion populations. It is known that Si affects slag properties and aids in minimizing reoxidation of Al in the ladle. This affects the ability of the slag to capture inclusions, the desulfurization rate, and changes the morphology of the inclusions. This study showed various differences between Si-bearing grades and RSi grades. Historically, RSi heats typically have a tight compositional distribution of inclusions; however, it was seen that RSi heats can mimic Si-bearing heats when the S wt.% drops below a certain threshold. This threshold was determined to be 0.003 wt.%. After the heat reached below this level, a compositional tail formation occurred in the inclusion distribution. This visually portrayed what a Si-bearing heat typically looks like even when the sulfur threshold has not been met. Si-bearing heats tend to desulfurize faster and to lower levels of sulfur; therefore, longer formations are not unusual on these heats. However, when looking into the FeSi addition timing versus the tail formation, it was noticed that the residual Ca in the trim addition of FeSi after desulfurization plateaued had a greater effect on the tail formation than the sulfur threshold value. Also it was noted that the longer the rinse time in the ladle after the threshold is met, the longer the tail formation was on both RSi and Si-bearing grades. FactSage software depicts Ca pickup in the steel from the slag.
to be very quick. However, it was determined that Ca pickup in the steel is directly related to the S wt.% and Si wt.% present in the steel. After the sulfur has reached below the threshold, it stops acting as a barrier to the slag, and ultimately drives the Ca out of the slag for inclusion modification.

Acknowledgments

The authors would like to thank the LMF team at Nucor Steel Tuscaloosa Inc. for their time and efforts put into getting the trials completed, as well as Maggie Saylor for her time in SEM analysis.

References