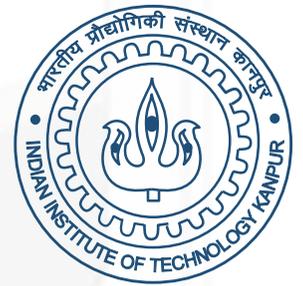


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# FACTORS AFFECTING THE EVOLUTION OF INCLUSION POPULATIONS DURING STEELMAKING AND CASTING PROCESSES

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**Abstract:** Investigations into the evolution of inclusion populations during ladle processing and continuous casting are reviewed. Details of the steelmaking practices employed in BOP and EAF steelmaking have been observed to have a strong influence on the size distribution, quantity, composition, phases and morphology of the final inclusion population delivered to the caster. Examples of the effects of different processing paths on the evolution of inclusion populations are presented. The importance of preexisting inclusion populations on the formation and removal of new inclusion populations is also reviewed and discussed.

**Keywords:** Steelmaking, Inclusions, PDF Analysis, AFA Analysis, Clean Steel

## 1. Introduction

The evolution of an inclusion population in the ladle is influenced by many factors, such as the type and size distribution of the parent inclusions in the steelmaking vessel at tap, the level of oxygen at tap, the amount of slag carryover from the steelmaking vessel, the type, quantity and timing of synthetic slag additions to the ladle, the type and timing of deoxidant additions and the timing and intensity of stirring in the ladle to name a few. Through plant studies and laboratory experiments, an understanding of how some of these factors affect the inclusions characteristics is discussed. Different heats were followed, and samples were taken at different times and stages of the steelmaking process.

## 2. Sampling procedure and analysis

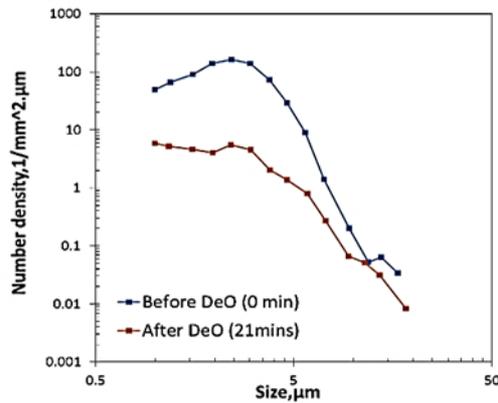
Immersion lollipops were taken at different times and locations during steelmaking. In lab studies, immersion and vacuum pin samplers were used. To minimize the presence of MnS inclusions formed during slow cooling, the sample surface was mounted and polished to a 1 $\mu$ m finish using diamond paste. Inclusion analysis was performed using the Aspex Pica 1020 SEM with EDS capability. Scans were performed at a magnification of 750X and a step size of 0.42 $\mu$ m. To represent the inclusion composition, a joint ternary plotting method with color coding was employed. Using this method, each ternary segment represents a distinct inclusion composition and hence, the whole inclusion population can be viewed in a single diagram.

## 3. Factors affecting the Inclusion Characteristics

### 3.1 Pre-Existing Inclusions and Method of Deoxidation

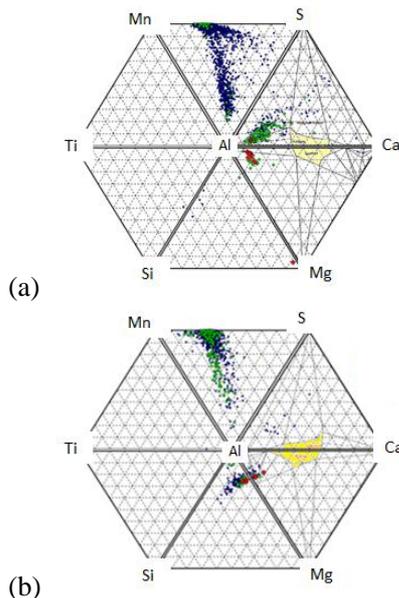
Deoxidation normally drives the first stage of inclusion evolution in the ladle. Aluminum, manganese, and silicon are typical deoxidants because of their strong affinity for oxygen, low dissolved oxygen residual, and formation of insoluble compounds <sup>[1]</sup>. Al<sub>2</sub>O<sub>3</sub> and MnO·SiO<sub>2</sub> inclusions are solid and liquid respectively at 1600°C. Due to its liquid nature, MnO·SiO<sub>2</sub> is normally spherical while the shape of Al<sub>2</sub>O<sub>3</sub> varies with oxygen levels at tap <sup>[2]</sup>.

The size and shape of the starting inclusion population in the ladle varies greatly depending upon the steelmaking practice. Pre-existing MnO·SiO<sub>2</sub> inclusions have been observed to produce spherical Al<sub>2</sub>O<sub>3</sub> inclusions <sup>[2]</sup>. In an EAF mini-mill study, similarities in the size distribution before and after deoxidation, shown in Figure 1, suggest that inclusion size after deoxidation is controlled by the size of the pre-existing inclusions. In this study, the steel was tapped at ~1000ppm and SEM analysis showed that the steel contained primarily FeO inclusions before deoxidation. After deoxidation, Al<sub>2</sub>O<sub>3</sub> inclusions were observed and both the Al<sub>2</sub>O<sub>3</sub> and FeO inclusions exhibited a similar population density shape and size distribution. The overall lower number density of the Al<sub>2</sub>O<sub>3</sub> inclusions is the result of inclusion agglomeration and flotation.



**Figure 1** Size distribution of FeO inclusions before and Al<sub>2</sub>O<sub>3</sub> inclusions after deoxidation (Figure shows similarities in both shape and size range)

Apart from inclusion shape and size distribution, the inclusion composition can also be different from what would be expected from equilibrium calculations. In two different mini mill plants, samples analyzed after aluminum deoxidation showed that plant ‘A’ contained calcium aluminates and plant ‘B’ contained Al-Mg oxide spinels. The inclusion composition for both samples is shown in Figure 2. In plant ‘A’, lime rich synthetic slag additions and aluminum were added at the same time after EAF tap while in plant ‘B’ deoxidant was added to the tap stream during EAF tap. With the kill-on-tap practice, the stream provides additional energy to promote the reduction of MgO from slag, promoting spinel formation. In a separate experiment where the practice was changed to killing after tap, Al<sub>2</sub>O<sub>3</sub> rather than Al-Mg oxide spinels formed.



**Figure 2.** Inclusion composition observed after deoxidation in (a) plant ‘A’, and (b) plant ‘B’ (Plant A contained mainly calcium aluminate and plant B contained magnesium spinels)

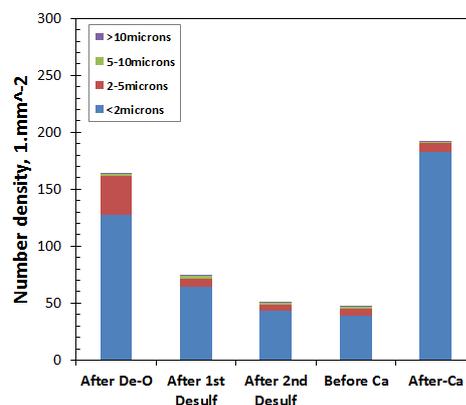
Removal of inclusions from liquid steel is dependent on the inclusions size and interfacial energy [3]. For similar inclusion types, larger inclusions will float out more readily than smaller inclusions. Also, the larger the interfacial energy, the more readily the inclusions agglomerate and float. Table 1 lists the interfacial energy for different inclusion types [4]. It suggests that Al<sub>2</sub>O<sub>3</sub> will float out more easily than other inclusion types. By controlling the inclusion composition and size after deoxidation, inclusions which readily float out can be generated and thus, cleaner steel can be produced.

**Table 1** Interfacial Energies for Different Inclusion Types in Steel

Inclusion	Interfacial Energy (erg/cm <sup>2</sup> )
Al <sub>2</sub> O <sub>3</sub>	2290
MnO·SiO <sub>2</sub>	1000
CaO· Al <sub>2</sub> O <sub>3</sub>	1300

### 3.2 Steel Grade and Ladle Stirring

Desulfurization is carried out at the LMF for steels produced by the EAF steelmaking route. During this process, the steel is mixed with a low oxygen, MgO saturated, lime rich slag by stirring with argon gas. Although desulfurization is normally not practiced for steel produced by BOP, argon stirring is still performed because it aids inclusion removal via bubble attachment. Figure 3 shows the change in inclusion content with time at the LMF of an EAF mini-mill. The reduction in inclusion number density before calcium treatment is due to argon stirring and inclusion removal.



**Figure 3** Reduction in number density with time due to argon stirring in a mini mill

In aluminum killed steel, transformation from alumina to Al-Mg oxide spinel occurs during desulfurization. This is due to MgO reduction from the slag by aluminum [5]. In high manganese, silicon containing steel grades,

transient manganese spinels have also been observed at the LMF in plant studies. Figure 4 shows the inclusion composition for a sample taken during production in a BOP steelmaking facility. It shows the presence of both silicates and Al-Mn spinels during stirring at the LMF. With time, transformation from Al-Mn oxide spinels to Al-Mg oxide spinels was observed, but that the reaction was very sluggish. Some heats still contained significant amounts of Al-Mn oxide spinels in final LMF samples. Figure 5 shows the phase diagram for the MnO-MgO-Al<sub>2</sub>O<sub>3</sub> system calculated using FactSage 6.4 and the FactPS, FTMisc, and FToxid databases. It shows that some cross-solubility exists between the Al-Mn and Al-Mg spinel phases. Depending on the local composition, both inclusion types can be stable.

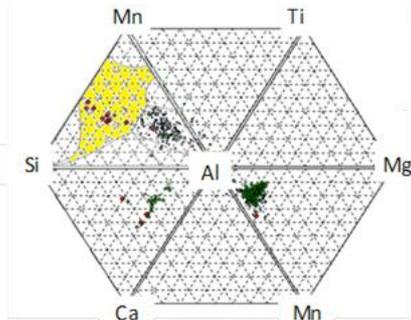


Figure 4 Inclusion composition showing presence of manganese aluminates at a BOP facility

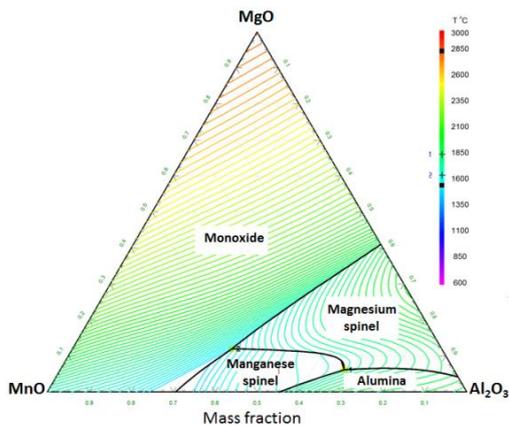


Figure 5 Phase diagram of the MnO-MgO-Al<sub>2</sub>O<sub>3</sub> system

### 3.3 MgO content in slag, reoxidation, and presence of CaS inclusions

Calcium is added to the LMF to modify existing solid alumina and spinel inclusions to liquid inclusions [6]. These liquid inclusions do not clog submerged entry nozzles (SEN), and are spherical in shape. Both alumina and Al-Mg oxide spinels can be modified by calcium.

However, CaS and MgO rich inclusions have also been observed in plant studies after Ca treatment. In Figure 3, an increase in the number density of inclusions after calcium treatment was observed. This is due to formation of CaS inclusions. These inclusions are solid at 1600°C and can clog SEN's. Figure 6 shows the result of a thermodynamic calculation using FactSage 6.4. The calculation was done at 1600°C for a typical line pipe steel grade containing Al-Mg oxide spinels. The results of the calculation shows that CaS inclusions can be formed from excess calcium additions. The formation of MgO inclusions during calcium modification is also predicted. MgO inclusions have also been observed in industrial samples. FactSage calculations with low initial Al-Mg oxide spinel content did not predict MgO inclusions after Ca treatment.

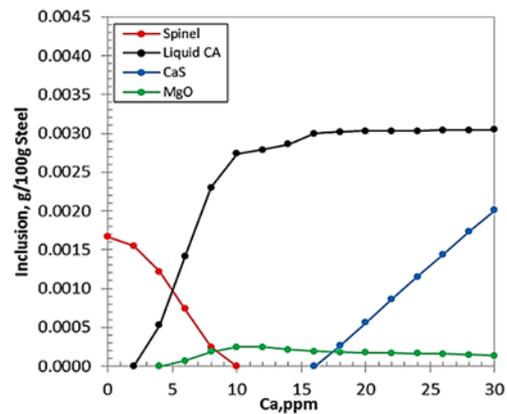


Figure 6 Thermodynamic calculation showing the formation of MgO during calcium modification of MgO saturated spinel

Steel reoxidation has been observed during ladle transfer in mini mills, resulting in changes from liquid calcium aluminates to solid calcium aluminates, alumina and spinel [7]. In the example shown in Figure 7, a plot of the area fraction and inclusion size for samples taken at different times in a laboratory study is shown. In this study, the steel was alloyed with manganese and silicon and then deoxidized with aluminum. Due to reoxidation in the experiment, the Al<sub>2</sub>O<sub>3</sub> inclusions formed after the Al addition gradually transformed to MnO·SiO<sub>2</sub> inclusions as the aluminum was depleted and Al<sub>2</sub>O<sub>3</sub> agglomerated and floated from the system. It is interesting to note that the population of the MnO·SiO<sub>2</sub> inclusions that formed after the aluminum was depleted were much smaller and appeared to nucleate independently from the pre-existing Al<sub>2</sub>O<sub>3</sub> inclusions.

In mini-mill samples taken at LMF final and from the tundish at different times during the startup of the 1<sup>st</sup> heat in a sequence, the presence of CaS inclusions has been observed to act as a source of calcium to modify inclusions formed by reoxidation. The proposed reaction mechanism is given by equations 1 and 2.

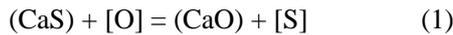
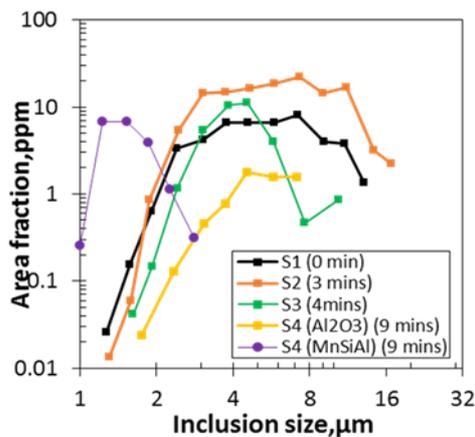


Figure 8 shows the inclusion composition for a sample taken after 90% of the steel had been transferred to the tundish. In tundish samples taken early after startup, evidence of reoxidation is normally observed by the presence of Al rich or unmodified spinel inclusions. However, as observed in this sample, the inclusions present are still fully modified liquid calcium aluminates. An increase in the amount of MnS inclusions was also observed, indicating S has transferred to the steel.



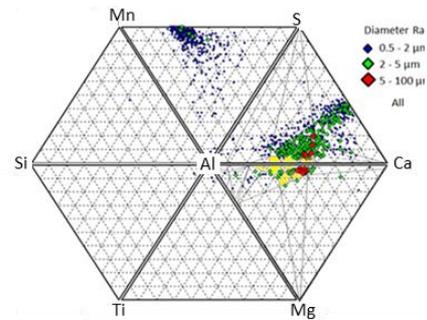
**Figure 7 Change in inclusion composition and size distribution resulting from steel reoxidation**

#### 4. Conclusion

Through plant and laboratory studies, the effects of different steelmaking practices on inclusion evolution have been presented and discussed.

- After deoxidation, the inclusion size distribution, shape and composition depends on pre-existing inclusions and the method and timing of deoxidation.
- Depending on steel chemistry, transformation of alumina to Al-Mg or Al-Mn oxide spinels can occur.
- Excess Ca additions during calcium treatment can produce CaS and increase the inclusion content. Depending on steels Mg content, MgO rich inclusions can be produced after Ca treatment.

- Liquid steel reoxidation changes both the inclusion size and composition. CaS formed after calcium treatment can act as a source of Ca to modify newly formed reoxidation inclusions.



**Figure 8 Inclusion composition during ladle transfer (Modified calcium aluminates are observed and result from CaS reaction with newly formed inclusions after reoxidation)**

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#### References

1. C.E. Sims and C.W. Briggs: Journal of Metals, December 1959, p.815.
2. M.-A. V Ende: Formation and Morphology of Non-Metallic Inclusions in Aluminum Killed Steel, Department of Materials Science, Katholieke Universiteit Leuven. 2010, P. 261.
3. L.A. Luyckx, Inclusion Removal, Metserv, Inc.
4. H. Suito and H. OHTA: ISIJ International, Vol. 46, 2006, p.31.
5. O. Adaba, R.J. O'Malley, S. N. Lekakh, V. L. Richards, and N. Sutcliffe: Proc., 9<sup>th</sup> international Conference and Exhibition on Clean Steel, Budapest, Hungary. 2015, chapter 4, paper 5.
6. N. Verma, P.C. Pistorius, R.J. Fruehan, M.S. Potter, H.G. Oltmann, E.G. Pretorius: Metallurgical and Materials Transactions B, Vol. 43B, 2012, p.830.
7. O. Adaba, P. Kaushik, R.J. O'Malley, S.N. Lekakh, V.L. Richards, E. Mantel, R. Hall, and E. J. Ellis: Iron & Steel Technology, July 2017, p. 38.