International Journal of Science and Research (IJSR)
ISSN: 2319-7064
Index Copernicus Value (2016): 79.57 | Impact Factor (2017): 7.296

Characterization of Inclusions during the Production of Stainless Steel with Focus on Submerged Nozzle Clogging

Eric Kojo Kweitsu[1], Asumadu Tabiri Kwayie[2], Ofori Yaw Nyantakyi[3]

[1]Mechanical Engineering Department, Koforidua Technical University, P. O. Box KF 981, Koforidua

[2]Materials Engineering Department, Sunyani Technical University, P. O. Box 206 Sunyani, Ghana

[3]Materials Engineering Department, Sunyani Technical University, P. O. Box 206 Sunyani

Abstract: ***Submerged Entry Nozzle (SEN) clogging is a problem encountered by a local steel industry during continuous casting of High Silicon Non Calcium treated (HSiNC) stainless steel. As part of solving this problem, inclusions were characterized during the production of the stainless steel. Samples were taken from two stainless steel heats (clogging Fe-23CR-19Ni-Si and non clogging Fe-19CR-12Ni-2Si) during ladle treatment and continuous casting. After sample preparation, inclusions characteristics (size, morphology, composition and number) were investigated using the 2D cross-sectional analytical method. The acquired inclusion information was then used to explain the reason for SEN clogging. Spinels (MgO-Al_2O_3) were the dominant inclusions found in the clogging heat and they formed clusters in the tundish during continuous casting. The solid spinel clusters blocked the submerged nozzle during continuous casting leading to SEN clogging. Calcium-Silicate-Magnesia-Aluminates (Ca-Si-Mg-Al)-O spherical inclusions were the dominant inclusions found in the non clogging heat. These inclusions were found to be liquid inclusions and therefore did not cause clogging.***

Keywords: stainless steel, inclusion, cluster, morphology, clogging

1. Introduction

The ever increasing demand for high quality steel products has necessitated the production of clean steels. Clean steel is not steel which does not contain impurities but rather the impurities are minimised to the lowest possible level. Production of steels with this level of quality requires high technical and capital input; as a result the total amount of clean steel produced in the world is comparatively small to crude steel [3].

The residual impurity elements commonly found in steel are oxygen, sulphur, phosphorus, hydrogen, nitrogen and even carbon. Oxygen and sulphur are responsible for the formation of oxide and sulphide inclusions which often affect the mechanical properties of steel. The presence of large amounts of oxide and sulphide inclusions decreases the ductility and fracture strength of steel products [5]. Fatigue, creep and impact test have shown that inclusions nucleate voids which grows into cracks and finally results in the fracture of steel material when a critical length of crack is exceeded [6]. Inferior surface appearance, poor polishability, and reduced resistance to corrosion are caused by large exogenous inclusions [7]. Inclusions also reduce the resistance of most steel products to hydrogen-induced cracking [8]. Studies have shown that most fatigue problems in steel are caused by hard and brittle oxide inclusions [4].

Apart from inclusions affecting the mechanical properties of steel products, solid inclusions can be deposited in the inner section (refractory linings) of submerged entry nozzle (SEN) of tundish and runners. The deposited inclusions block the nozzle and eventually stop the flow of liquid steel during casting. This problem is called Submerged Entry Nozzle (SEN) clogging, when partial clogging occurs during continuous casting, the stopper in the tundish can be raised and casting continues. However in the case of full clogging, continuous casting must be stopped and the steel in the ladle is carried back to the ladle station whiles the liquid steel in the tundish become waste. The interferences in the production process lead to the loss of time and energy. SEN clogging can also be very detrimental to steel cleanliness because dislodged clogs get trapped in the liquid steel and form large exogenous inclusions. This aside, clogs trapped in nozzle can change the flow pattern and jet characteristics already existing in the nozzle, which turns to disrupt flow into the mould resulting in slag entrainment and surface defects [2].

It is obvious from the above discussion that the effect of inclusions cannot be under estimated. It is therefore important to control the steel making process to minimize the amount of inclusions generated in the steel. The problem of Submerged Entry Nozzle (SEN) clogging as discussed above was encountered in a local steel industry during the continuous casting of High Silicon Non- Calcium treated (HSiNC) stainless steel. As part of solving this problem, inclusions characteristics (Size, number, morphology and composition) from two stainless steel heats (clogging Fe-23CR-19Ni-Si and non- clogging Fe-19CR-12Ni-2Si) were investigated and compared using the two dimensional (2D) cross-sectional analytical method. The acquired inclusion information was then used to explain the reason for SEN clogging.

1.1 Aim

The main aim of this project is to detect and evaluate macro and micro inclusions in stainless steel grades

1.2 Objectives

The objectives of this study includes:
- Classification of inclusions according to composition
- Evaluate inclusions according to morphology
- To identify and study the physical characteristics of the inclusions
- To evaluate the quantity of inclusions in clogging and non clogging heat.

1.3 Scope

The scope of the research was limited to stainless steels.

1.4 Methodology

This study made use of qualitative analysis for the experimental work and qualitative analysis for the analysis of secondary data.

2. Experimental Procedure

In this study, two high-silicon non-calcium treated (HSiNC) stainless steel grades of similar composition were selected, one is a clogging heat (Fe-23CR-19Ni-Si) and the other is a non clogging heat (Fe-19CR-12Ni-2Si). The elemental content of the two steel grades is given in Table 1. Four lollipop samples were taken from each heat as shown in fig.2.1. The first sample (L1) was taken at the start of ladle treatment and the second sample (L2) was taken at the end of ladle treatment. The third (T1) and fourth (T2) samples were taken at the onset of continuous casting and thirty minutes after continuous casting respectively.

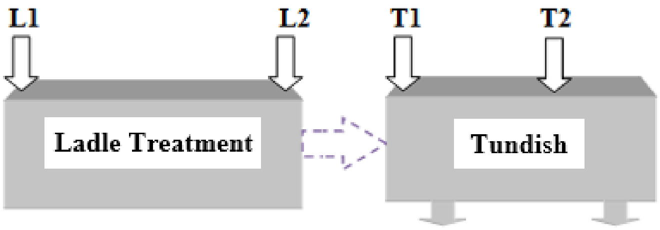


Figure 2.1: Sampling location and moments

The middle zone of the lollipop samples were cut according to the dimension 20mm x 12mm x 8mm as shown in fig.2.2 below. The samples were then prepared and by the help of the Scanning Electron Microscope (SEM) equipped with Energy Dispersive X-ray Spectroscope (EDXS), qualitative and quantitative assessment of inclusion characteristics were carried out.

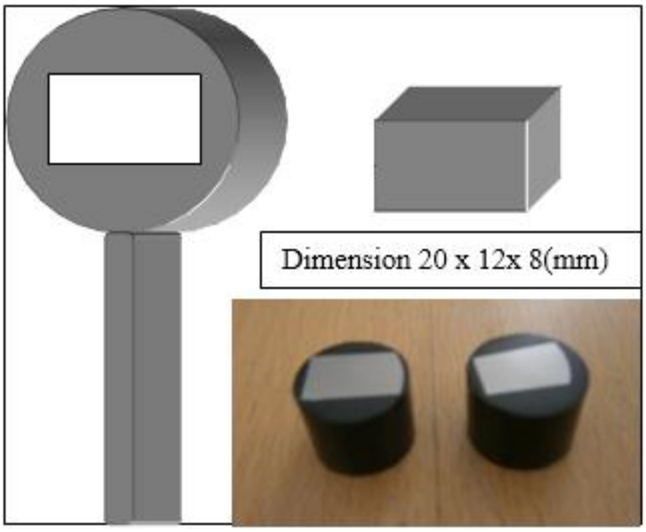


Figure 2.2: Sample preparation and samples after polishing

The qualitative assessment was done to investigate inclusion composition and morphology whiles quantitative assessment was used to investigate the number and size of inclusions. Inclusions were characterised according to their elemental composition, at least 20 inclusions were analysed per each sample from both heats.

Table 1: Chemical Composition of Steel grades

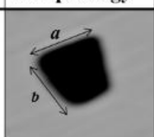
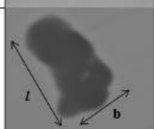
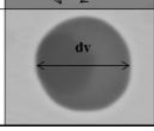
Steel Grade	Chemical Composition (mass %)									
	C	Si	Ni	Mo	Cr	Mn	P	S	N	Al
Clogging (C)	0.021	2.68	19.16	0.26	23.36	1.72	0.024	0.006	0.043	0.003
Non Clogging (NC)	0.053	1.88	11.69	0.60	19.33	1.14	0.024	0.001	0.027	0.003

3. Results and Discussion

3.1Classification of inclusions according to composition

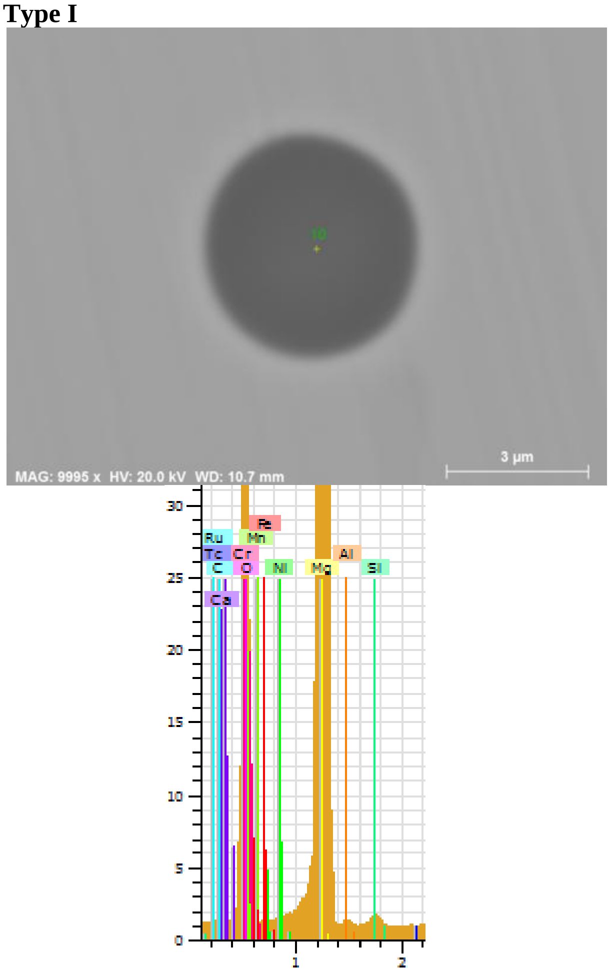
In this study, inclusions in both clogging and non clogging heats were classified into three basic types (Type I, II and III) based on composition as shown in table 4.1. The Type I inclusions were Periclase (MgO) having particle size ranging from 2-4µm. They were found in both heats at the start of ladle treatment.

Table 4.1 Types and composition of inclusions observed

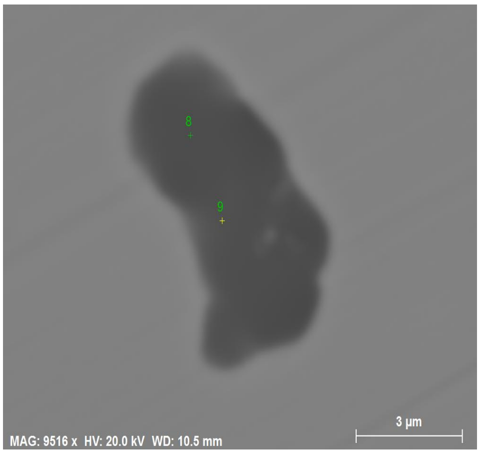
Type	Composition(wt%)	Morphology	Size = $\sqrt{Area_{max}}$
I	**Periclase** MgO		$\sqrt{a \times b}$ 2-4µm
II	**Spinel** MgO - Al_2O_3- (MnO,SiO_2,CaO) (65-75) (20-25) (5-10)		$\sqrt{l X b}$ 3-7µm
III	**Calcium-silicate-magnesia-Aluminates(Ca-Si-Mg-Al)-O)** (15-50) (25-35) (20-35) (5-15) CaO SiO_2 MgO Al_2O_3		$\sqrt{\Pi} \times \frac{dv}{2}$ 2-8µm

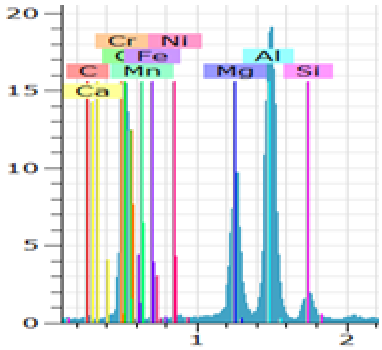
The Type II inclusions were spinel ($MgO-Al_2O_3$-(MnO, SiO2, CaO) inclusions, its size ranged from 3-7µm and were found only in the clogging heat. Calcium-Silicate-Magnesia-Aluminates (Ca-Si-Mg-Al)-O) inclusions were classified as Type III, the particle size ranged from 2-8µm and were found only in the non clogging heat. Fig. 4.1 shows the EDXS spectrum of the various types of inclusions.

(a) Type I

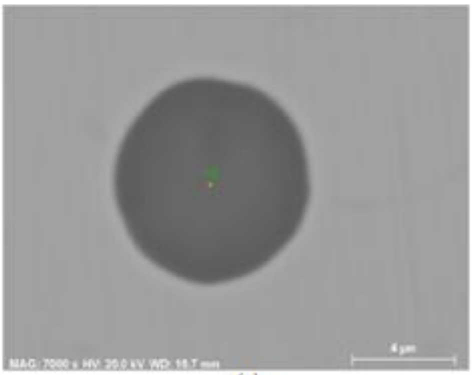


(b) Type II





(b) Type III



(c)

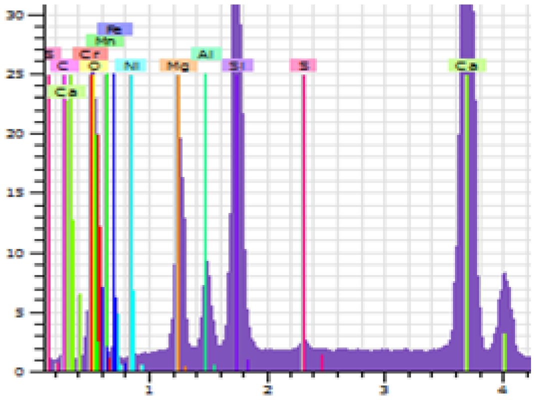


Figure 4.1: Typical EXDS spectrum of (a) Type I (MgO), (b) Type II (Spinel) and (c)Type III ((Ca-Si-Mg-Al)-O inclusion)

4.1.1 Quantity of inclusions in Clogging and Non clogging heat

The number of the different types of inclusions observed during each stage of production is represented in fig, 4.2. The solid and dash lines denote the clogging heat(C) and the non clogging heat (NC) respectively. From fig. 4.2 (a) it is obvious that high amount of MgO inclusions (Type I) were observed in both heats at the start of ladle treatment, about 50% of inclusions analysed at this stage of production were MgO. The actual number of MgO inclusions counted as shown by fig.4.2 (b) was 12 and 13 in the clogging heat and non clogging heat respectively.

These MgO inclusions were not generated in the ladle but were carried over from the AOD into the ladle. From the first graph of fig.4.2 (a), it appears that most MgO inclusions were removed out of the clogging heat after ladle treatment. The explanation for this is that most of the MgO inclusions in the clogging heat were transformed into spinel (type II) during ladle treatment as evident in the second graph of fig.4.2 (a). There was a significant increase in the number of spinel counted at the end of ladle treatment from 4 to 17 as shown in the second graph of fig4.2 (b), this remained nearly the same around 18 and 20 during casting.

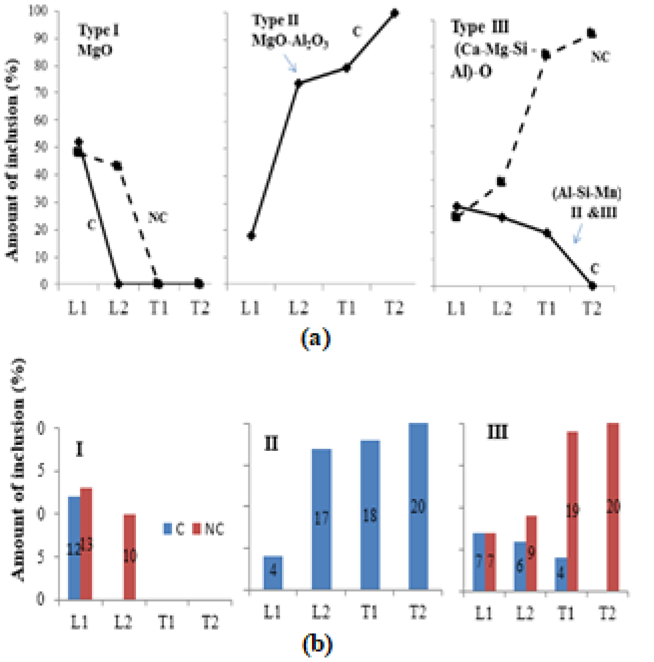


Figure 4.2: Amount and types of inclusions in both heats (a) amount of inclusion in percentage (b) actual number of inclusion.

The spinel is a typical product of deoxidation and was formed as a result of high content of aluminium coupled with high quantity of MgO in the liquid steel at the start of ladle treatment. Historically the earliest finding of spinel was documented in the 1980's where spinel was observed in 301 stainless steels [9]. Spinel is reported to have quite similar behaviour as alumina inclusions; it has relatively high melting point and high hardness. It has also been identified as a potential cause of nozzle clogging as well as defects in steel products [9].

Most of the MgO inclusions in the non clogging heat were transformed to Type III as shown by the third graph of figure 4.2(a). Although the steel was not calcium treated, the type III had calcium content ranging between 15 to 50wt%. The possible sources of calcium are the slag system as shown by fig.4.3 and ferrosilicon. Ferrosilicon used for deoxidation normally contains about 1% calcium and aluminium each [9] and this introduces calcium and aluminium into the liquid steel.

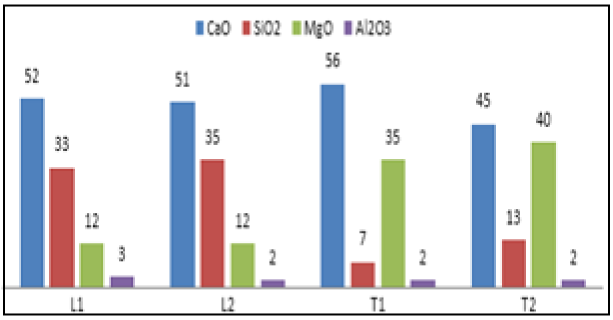


Figure 4.3: Slag composition for non-clogging heat

4.2 Classification of inclusions according to morphology

The inclusions in both heats were further classified according to their morphology that is regular, irregular and spherical inclusions. Fig 4.3 shows the amount of inclusions with different types of morphology classified during each stage of production.

The Type I inclusions (MgO) mostly had regular morphology which transformed into regular shaped spinel (Type II) in the clogging heat during ladle treatment. The spinel then formed clusters with irregular morphology during continuous casting as illustrated by fig 4.4.

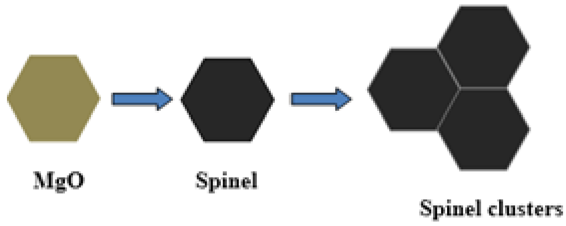


Figure 4.4: Spinel morphology

More spinel clusters were formed in the tundish during continuous casting, this accounted for the high amount of irregular inclusions observed in the clogging heat as shown by the first graph of fig. 4.3 (T1 and T2). During casting, the temperature of the liquid steel in the tundish begins to drop, the spinel inclusions therefore coagulate to form cluster. Some of the spinel clusters observed in the tundish are shown in fig. 4.5, the presence of this spinel cluster in the liquid steel during continuous casting led to clogging of the submerged nozzle. The Type III inclusions observed in the non clogging heat had spherical morphology, the third graph of fig. 4.3 shows that the spherical inclusions become dominant in the liquid steel during casting.

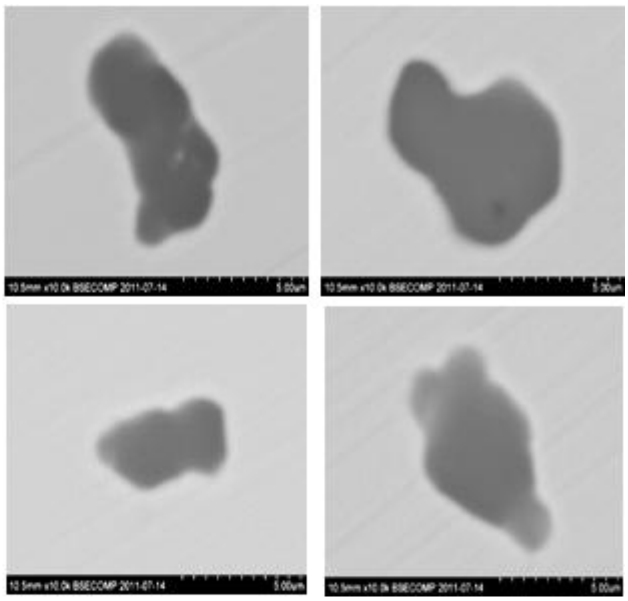
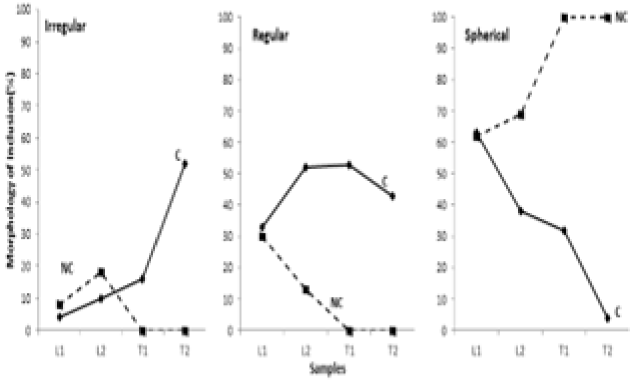


Figure 4.5: Examples of spinel clusters found in the tundish

Paper ID: ART201849 DOI: 10.21275/ART201849

ISSN: 2319-7064
Index Copernicus Value (2016): 79.57 | Impact Factor (2017): 7.296

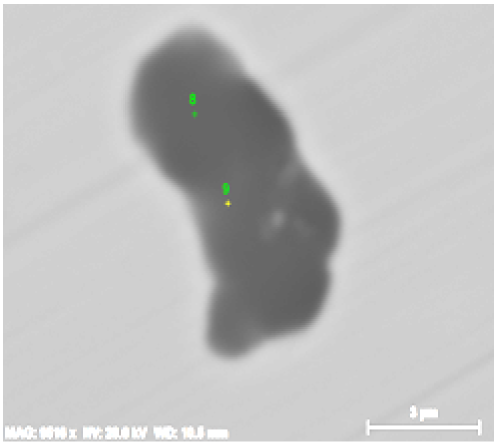


4.3. Physical characteristics of inclusions

1) Type I MgO

The EDXS analysis shows that the spinels observed in the clogging heat were composed of 20-25wt%MgO and 65-75wt% Al_2O_3 with traces of 5-10wt% MnO, SiO_2 and CaO. Referring to the corresponding phase diagram fig.4.6, the composition of the analysed spinel particle varies along the red arrow in the phase diagram. At this composition the spinel particle is solid at temperatures below 2135^{o}C; this means that even at the steel making temperature (1700^{o}C), the spinel particle remains in its solid state.

The continuous deposition of the solid spinel particles in the inner section of the nozzle will eventually block it, stop the free flow of liquid steel and lead to the problem of SEN clogging. Spinel inclusions do not only bring about SEN clogging but also has the potential to cause sliver defects on surfaces of hot and cold rolled steel sheets. [9]



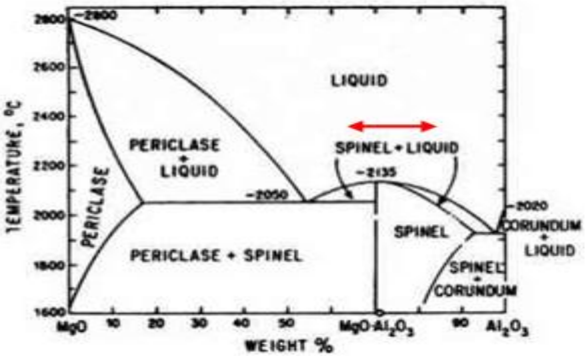


Figure 4.6: Phase diagram of MgO and Al_2O_3 [3] arrow in phase diagram showing composition of spinel

2) Type III Calcium-Silica-Magnesia-Aluminates

The (Ca-Mg-Si-Al)-O inclusions behaved like a system of inclusion, having its composition similar to the slag system used during ladle treatment. Fig.4.7 shows how the oxides of this system of inclusion varied during ladle treatment and continuous casting. It is obvious that all the (Ca-Mg-Si-Al)–O inclusions analysed during ladle treatment were composed of high calcium oxide with lower amount of alumina. The MgO and SiO_2 content increased considerably during steel refinement to continuous casting.

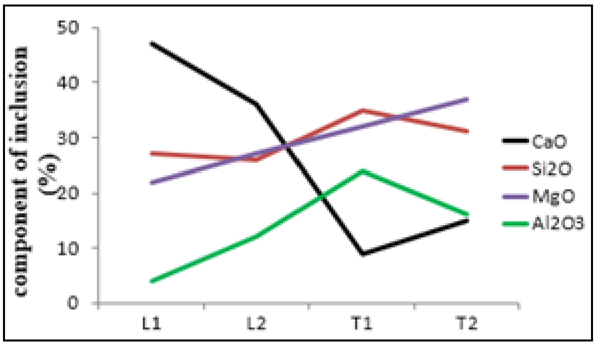


Figure 4.7: Variations in the oxide composition of (Ca-Si-Mg-Al)-O inclusion

When the composition of (Ca-Si-Mg-Al)-O inclusion was plotted on a ternary phase diagram of CaO, SiO and MgO at 10 wt% Al_2O_3 as shown in fig.4.8, it was observed that the inclusions were in the liquid state during casting. The composition of the inclusions during ladle treatment (L1 and L2) shifted to the liquid region during casting (T1 and T2) as indicated in the phase diagram. Liquid inclusions cannot block the submerged nozzle and therefore do not cause clogging. A detailed analysis of the link between inclusion composition and casting behaviour revealed that clogging can be avoided if the inclusions contain more than 50% liquid [10].

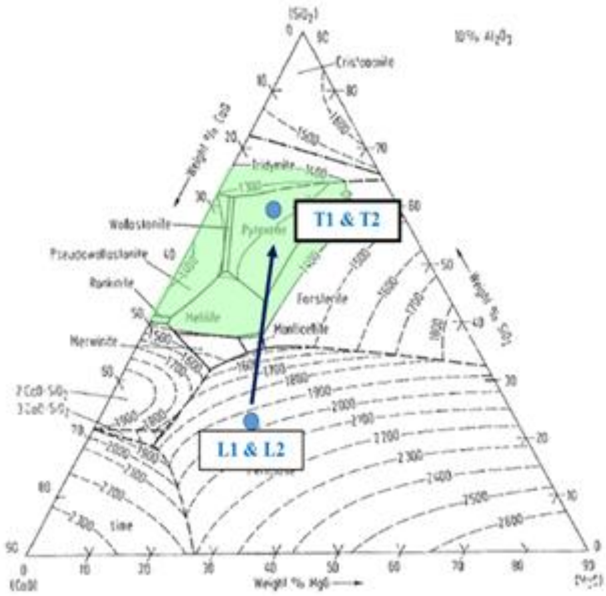


Figure 4.8: Ternary phase diagram for CaO, SiO2 and MgO at 10wt% Al_2O_3 mashed area showing liquid zone [1]

This is what calcium treatment of clogging steel heat seeks to achieve. When inclusions contain much liquid it become virtually impossible to block the submerge entry nozzle.

4.4 Formation Mechanism of Spinel

The theory for formation of singular spinel consisting of only $MgO.Al_2O_3$ during steel production is discussed below. Fig.4.9 shows the proposed mechanism for spinel formation in stainless steel commonly presented in literature. The ladle slag system employed during ladle treatment for the clogging heat is typically composed of 57% CaO, 28% SiO2, 9% MgO and 2% Al_2O_3. The total amount of FeO, MnO and CaF in the ladle slag was below 4%.

The formation of spinel begins with the supply of Mg into liquid steel, since there is no addition of Mg during the steel making process; the only source of Mg is from the reduction of MgO from the refractory or the slag. When equilibrium is maintained between the molten steel and inclusion, soluble Mg is supplied into the liquid steel by the reduction of MgO with aluminium in the steel. The reduction reaction is illustrated below;

$3(MgO)_{\text{in slag}} + 2\underline{Al} = (Al_2O_3)_{\text{in slag}} + 3\underline{Mg}$........................ (a)

$3MgO(s)_{\text{in refractory}} + 2\underline{Al} = (Al_2O_3)$ in slag $+ 3\underline{Mg}$........... (b)

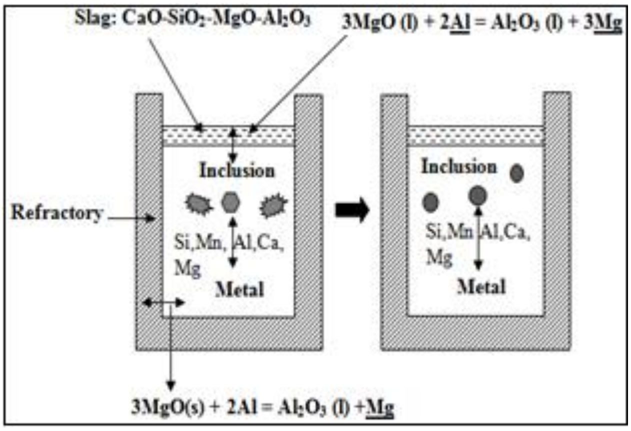


Figure 4.9: Schematic illustration of the mechanism for formation of spinel inclusions

Since diffusion is much faster in liquid than solids, the supply of Mg from the liquid slag (reaction a) is more dominate than from the solid refractory (reaction b). It is also very important to take into account the dissolution of MgO from refractory into the slag phase as shown by reaction(c) as another source of Mg in the liquid steel.

$MgO(s)_{\text{in refractory}} = (MgO)_{\text{in slag}}$.......................................(c)

When the steel is killed with aluminium, alumina inclusions are formed as soon as Al is added into the molten steel. The alumina inclusions then react with the soluble Mg leading to the formation of MgO.Al2O3 spinel inclusions. This reaction is illustrated below;

$4\,Al_2O_{3(s)} + 3\underline{Mg} = 3MgO.Al_2O_{3(s)} + 2\underline{Al}$.................... (d)

The formation of spinel by reaction (d) is the simplest case where alumina is the primary inclusion formed when the steel is deoxidised with Al. For silicon "killed" steel which results in the formation of silicate inclusions the situation is more complex. The major reaction that takes place is as follows;

$SiO_2\,(l) + 2Mg = 2MgO\,(l) + Si$.................................. (e)

$3SiO_2\,(l) + 2(Al) = 2Al_2O_3\,(l) + 3Si$........................... (f)

The MgO and Al_2O_3 inclusions then become concentrated in the liquid steel and they are gradually changed into spinel. Two main factors are responsible for transforming MgO and Al_2O_3 inclusions into spinel, these are the sources of aluminium and the activities of oxides in the slag. For aluminium "killed" steels, aluminium as a deoxidizer serves as the main source of aluminium in the liquid steel while in the case of silicon killed steel, aluminium found in the ferrosilicon (Fe-Si) serve as the main source of aluminium in the liquid steel. Research has shown that most Fe-Si used as deoxidizers contains at least 1wt % of Al and Ca each [9].

The two mechanisms for generation of spinel inclusions discussed above hold for the clogging heat. Although the steel was not "killed" with aluminium, the trace of aluminium introduced by the ferrosilicon was enough for the generation of spinel inclusion by reactions (d), (e) and (f) in the clogging heat. The amount of ferrosilicon used for deoxidising the clogging heat was more than the non clogging heat. This resulted in relatively high amount of silicon and aluminium in the clogging heat as shown by figure 4.10 and 4.11 at the start of ladle treatment. The high amount of aluminium coupled with high MgO content in the liquid steel at the start of ladle treatment is the main cause of spinel formation.

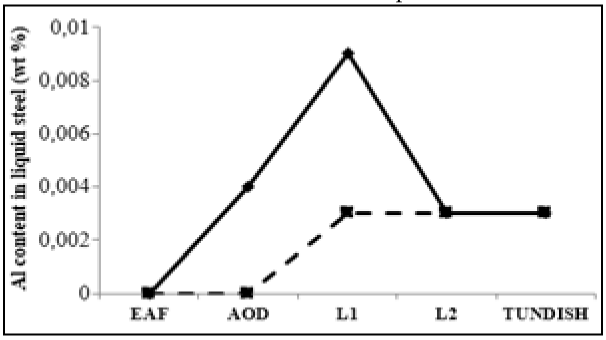


Figure 4.10: Aluminium content in clogging(C) and non-clogging heat (NC).

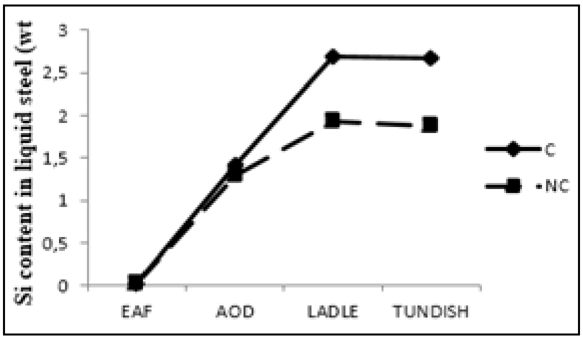


Figure 4.11: Silicon content in clogging heat(C) and non-clogging heat (NC)

The high amount of silicon in the clogging heat can also be attributed to the reduction of SiO_2 by aluminium introducing some silicon into the liquid steel. The presence of aluminium does not only reduce the amount of dissolved oxygen but can also increase the amount of silicon in the liquid steel.

4. Conclusion

Three types of inclusions were identified, Type I inclusions being MgO, Type II inclusions were spinels ($MgO-Al_2O_3$) and Type III inclusions were calcium-silicate-magnesia-aluminates (Ca-Si-Mg-Al)-O.

Type I inclusions (MgO) were observed in both heats at the start of ladle treatment, it transformed to spinels (Type II inclusions) and Calcium-silicate-magnesia-aluminates (Type III inclusions) in the clogging and non clogging heat respectively.

Type II inclusions ($MgO-Al_2O_3$) were the dominant inclusions found in the clogging heat; they formed clusters with size ranging from 3-7µm. The formation of spinel clusters begun in the tundish and became dominant in the liquid steel during continuous casting. The formation of high amount of spinel inclusions were attributed to the high content of aluminium coupled with the high amount of MgO inclusions in the liquid steel at the start of ladle treatment. The spinel clusters were solid in nature which is likely to get deposited in the nozzle during casting and caused SEN clogging.

The non clogging heat was mainly composed of Calcium-silicate-magnesia-aluminates (Ca-Si-Mg-Al)-O inclusions. They were formed during the waiting time for continuous casting. They had spherical morphology and were found to be liquid in nature and therefore did not cause clogging. Although the steel was not calcium treated, these inclusions contained high calcium which gave the inclusion its spherical morphology and liquid nature.

References

[1] S. Millman, Clean steel Basic features and operating practices, International Iron and Steel Institute, pp. 8-10, 2004
[2] Rob Dekkers, Characterization of Inclusion, 2002
[3] Steel Statistical Yearbook 2002, International Iron and Steel Institute, pp. 13-16. 2002
[4] E. Fuchs and P. Jonsson: High Temperature Mater. Proc. vol. 19 (5), pp. 333-43. 2000
[5] P.K. Trojan: ASM Handbook, vol. 15, Casting, ASM INTERNATIONAL, Metals Park, pp. 88-97. 1988
[6] T.J. Baker and J.A. Charles, Effect of Second-Phase Particles on the Mechanical Properties of Steel, The Iron and Steel Institute, pp. 88-94. 1971
[7] S.E. Lunner, Production and Application of Clean Steels, the Iron and Steel Institute, pp. 124-36, 1970
[8] A.D. Wilson, Advances in the Production and Use of Steel with Improved Internal Cleanliness, ASTM, pp. 73-88, 1999
[9] Joo Hyun Park and Hidekazu Todoroki, Control of MgO Al_2O_3 Spinel Inclusions in Stainless Steels, ISIJ International, Vol. 50 No. 10, pp. 1333–1346, 2010
[10] F. Fuhr, C. Cicutti, G. Walter and G. Torga, Relationship between nozzle deposits and inclusion composition in the continuous casting of steels, ISS Tech Conference Proceedings, pp 165-175, 2003

Author Profile

Eric Kojo Kweitsu is currently a Lecturer at the Mechanical Engineering Department of the Koforidua Technical University, Koforidua- Ghana. His Research Interests Includes: Process Metallurgy, Materials Design, Materials Failure Analysis and Welding Metallurgy. He holds an MSc in Materials Science and Engineering for the Royal Institute of Technology (KTH)- Sweden. He also holds a BSc in Materials Engineering form the Kwame Nkrumah University of Science and Technology.

Asumadu Tabiri Kwayie is a lecturer at the Materials Engineering Department of Sunyani Technical University, Sunyani – Ghana where he is currently the Head of Department. He holds a MSc. degree in Materials Engineering from the Royal Institute of Technology (KTH), Stockholm – Sweden and is a specialist in two fields of metallurgy which are Industrial Process Metallurgy and Materials Design. Tabiri did his BSc. degree in Materials Engineering at the Kwame Nkrumah University of Science and Technology.

Yaw Ofori Nyantakyi is an engineer, lecturer and an armchair philosopher. He's currently a lecturer at the Materials Engineering Department of Sunyani Technical University. His research interests are mainly in corrosion control and process control. His first degree is in materials engineering from KNUST,Ghana and a master's degree in oil and gas technology from Aalborg university in Denmark