ANALYSIS OF THE MORPHOLOGY OF NON-METALLIC INCLUSIONS IN CONTINUOUSLY CAST STEEL SEMIFINISHED PRODUCTS

Iulia-Olivia POENARU[1*], Miruna MAGAON[2], Ana SOCALICI[3], Adriana BOBORA[4]

The control of non-metallic inclusions is critical for ensuring steel cleanliness, improving mechanical performance, and preventing nozzle clogging during continuous casting. This study investigates the chemical composition, morphology, and structural characteristics of inclusions in three steel samples processed under vacuum conditions with aluminum and calcium deoxidation. Scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDAX) analyses. Prolonged inclusion formation in certain regions suggests incomplete deoxidation at the micro-scale. This research provides insight into inclusion evolution during secondary metallurgy and highlights the importance of precise control of Al, Ca, and process conditions to optimize inclusion modification and minimize casting defects.

Keywords: non-metallic inclusions, steel cleanliness, calcium treatment, deoxidation, SEM-EDAX analysis, continuous casting, inclusion morphology.

1. Introduction

Non-metallic inclusions—primarily oxides, sulfides, and complex compounds—are important in defining the quality of steel, affecting aspects such as mechanical performance, fatigue resistance, and castability. These inclusions form as a result of deoxidation, slag–metal reactions, refractory erosion, and microalloying practices [1]. In particular, alumina (Al_2O_3) and spinel ($MgO.Al_2O_3$) inclusions are notorious for causing nozzle clogging during continuous casting, leading to mold-level instabilities, surface defects, and productivity losses [2]. Among the techniques to improve inclusion behavior, calcium treatment is acknowledged as the most effective: it modifies solid alumina inclusions into more deformable, low-melting calcium aluminates, which favours the flotation in the slag [1]. However, its efficacy is highly dependent on precise control of calcium,

[1*] PhD Student, Dept. of Engineering and Management, University POLITEHNICA of Timisoara, Faculty of Engineering Hunedoara, Romania, corresponding author, e-mail: poenaruiualiaolivia@yahoo.com,
[2] PhD, Dept. of Engineering and Management, University POLITEHNICA of Timisoara, Faculty of Engineering Hunedoara, Romania, e-mail: miruna.magaon@yahoo.com
[3] Professor, Dept. of Engineering and Management, University POLITEHNICA of Timisoara, Faculty of Engineering Hunedoara, Romania, e-mail: virginia.socalici@fih.upt.ro
[4] PhD Student, Dept. of Engineering and Management, University POLITEHNICA of Timisoara, Faculty of Engineering Hunedoara, Romania, e-mail: gulasadriana@gmail.com

aluminum, and sulfur contents, the timing of additions, and coordination of post-treatment aluminum reintroduction, which can reverse inclusion modification efforts [3].

The recent studies [1-5] offer deeper insight into the thermochemistry and kinetics underlying inclusion evolution during secondary metallurgy, achieving liquid calcium aluminate inclusion phases and avoiding calcium sulfide formation—the latter being detrimental to steel fluidity and nozzle flow. At the same time, magnesium uptake from refractory erosion has been identified as a contributing factor to spinel formation, competing with calcium modification [5]. Moreover, experimental investigations highlight the influence of hydrodynamic conditions—such as argon stirring—on inclusion flotation and transport, impacting clogging behavior and steel cleanliness. The analyze of nozzle clogging mechanisms differentiates early-stage chemical precipitation from late-stage particulate blocking, reveals the importance of inclusion morphology and mobility in preventing clogging in aluminum-killed steels. The current experimental research further defines optimal calcium range where inclusion phases transition favorably toward liquid and semi-liquid domains, ensuring both inclusion modification and castability.

2. Experimental research

Experimental analyses were performed to determine the morphology and chemical composition of non-metallic inclusions within continuously cast St52.3A steel semi-finished products with diameter 280mm according EN 10025-2: 2005 (Table 1).

Table 1

Chemical composition of St52.3A corresponding SR EN

Elements	C(%)	Mn(%)	Si(%)	S(%)	P(%)	Cr(%)	Ti(%)	B(%)	Al(%)	N(ppm)	Ca(ppm)	Ti/N*
Min.	0.25	1.3	0.25	-	-	0.08	0.012	0.02	-	15	15	2.9
Max.	0.28	1.4	0.35	0.005	0.005	0.18	0.027	0.04	70	40	40	-

The billets were monitored across the complete process route (Figure 1), beginning with EAF steelmaking, where high-purity charge materials were used to minimize surface oxidation and contamination. Secondary refining in the LF and VD units was conducted under controlled thermochemical conditions, with moisture-free alloy additions and argon stirring ensuring homogeneous composition and temperature. Vacuum degassing was performed without supplementary heating, and all transfer vessels were preheated to preserve thermal consistency. During continuous casting, process stability was maintained through coordinated control of casting speed, steel superheat, and secondary cooling parameters, ensuring optimized solidification and defect prevention.

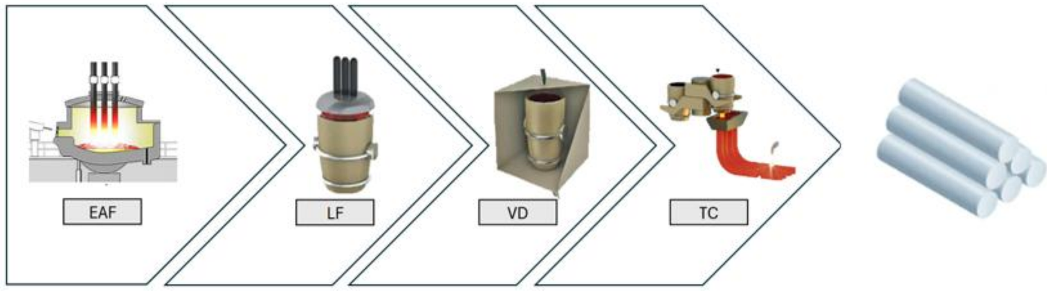

Fig. 1. Steel process stages

To ensure optimal deoxidation efficiency, a rigorously controlled ladle addition sequence was employed. Tapping was executed with full retention of the refining slag, after which lime was charged to initiate basic slag formation. Primary deoxidation was achieved through aluminum addition accompanied by the first carbon adjustment. Upon filling approximately one-quarter of the ladle volume, ferroalloys, supplementary carbon sources, bauxite, and desulfurizing agents were introduced. Final slag reduction was carried out near the end of tapping through the addition of ferrosilicon, aluminum, silicon carbide, and silico-calcium, ensuring a highly reduced and fully active refining slag.

To decrease the oxygen activity in the molten steel, the following deoxidation practice was implemented: an initial addition of 10 kg of aluminum at tapping (mandatory), followed by supplementary additions at a rate of 10 kg Al per 100 ppm [O_2]. During the entire ladle filling process, argon was injected through two porous plugs located at the ladle bottom, ensuring continuous bath agitation. This argon stirring promoted efficient deoxidation kinetics and facilitated the flotation and removal of reaction products.

Argon bubbling ensured proper emulsification and interaction between the slag and the metal bath, thereby accelerating chemical homogenization. Simultaneously, this process enhanced the separation of complex oxide inclusions from the steel into the slag phase, according to the generalized reaction (1):

$$[((Si - Al - Mn)_x O_y)]_{metal} \rightarrow [((Si - Al - Mn)_x O_y)]_{slag} \quad (1)$$

At this stage of the steelmaking process, the formation of a refining slag is of critical importance, as it performs several essential functions: promoting deoxidation and desulfurization, protecting the molten steel from reoxidation, absorbing non-metallic inclusions and retaining dissolved gases while enabling their controlled release into the atmosphere of the secondary refining aggregate.

In the ladle furnace, slag formation was promoted through argon stirring to emulsify the slag–metal interface. A lime–bauxite mixture was added in three stages to generate a basic slag with optimal refining capacity, while maintaining a slag mass of 15–20 kg/t to preserve steel cleanliness. Deoxidation was achieved using a strongly reducing mixture of carbonaceous materials and high-affinity deoxidizers (Al, SiC, FeSi, SiCa, AlCaSi), with proportions adjusted to favor oxygen removal and minimize oxide activity. Ferroalloys were added according to steel grade requirements, followed by a 2–3 minutes interval with intensive argon stirring to enhance chemical and thermal homogenization, sulfur removal, inclusion flotation, and hydrogen/nitrogen desorption. Corrective alloying was strictly limited in mass and timing to prevent late-stage oxide inclusions. Final microalloying with aluminum wire was performed under controlled immersion and brief argon bubbling to prevent reoxidation and ensure low residual non-metallic inclusion content. Thereafter, calcium treatment was conducted through the immersion of SiCa wire to promote the modification and spheroidization of alumina (Al_2O_3) inclusions, improving their morphology and preventing nozzle clogging during continuous casting. In accordance with best practice, aluminum wire additions were strictly prohibited after SiCa treatment to avoid reoxidation and undesirable inclusion morphology.

To achieve chemical and thermal homogenization of the molten steel in the ladle, as well as to facilitate the flotation and removal of final reaction products, gentle argon bubbling was applied for approximately 5 minutes following the addition of the SiCa-filled wire, while maintaining the steel surface fully covered to prevent reoxidation. Non-compliance with this operational requirement may result in significant meniscus fluctuations within the mold during continuous casting, leading to surface defects on the cast billets, clogging of submerged entry nozzles (SEN), and ultimately, unplanned interruption of the casting process.

Moreover, excessive SiCa additions can cause substantial precipitation of calcium sulfide (CaS), which adversely affects steel fluidity. This phenomenon can further amplify mold level fluctuations and aggravate SEN clogging, potentially culminating in complete nozzle blockage. Such occurrences negatively impact both the surface quality of semi-finished products and the overall productivity of the casting line

Three representative samples were collected from the experimental heats for detailed characterization of non-metallic inclusions. The inclusion morphology and chemical nature were investigated by microstructural examination using scanning electron microscopy (SEM) combined with energy-dispersive X-ray spectroscopy (EDAX) on a Quanta Inspect F system.

The EDAX spectrum for micro-area 1 shown in Figure 2 revealed the presence of C, O, Mg, Al, Si, S, Ca, and Fe (Figure 3, micro area 1), suggesting that the inclusion population consisted primarily of alumina-based oxides modified by Mg and Ca, possibly forming spinel-type inclusions and calcium aluminates. Such inclusions typically originate from the deoxidation sequence involving aluminum and subsequent modification by calcium introduced through SiCa wire feeding.

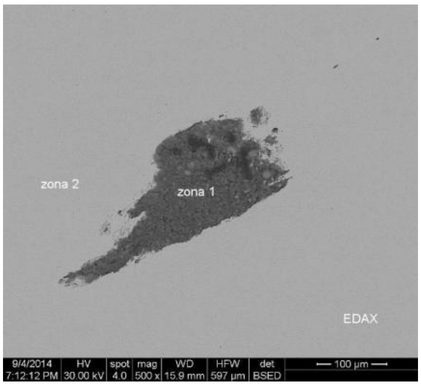

Fig. 2. EDAX Analysis, sample 1, 500x

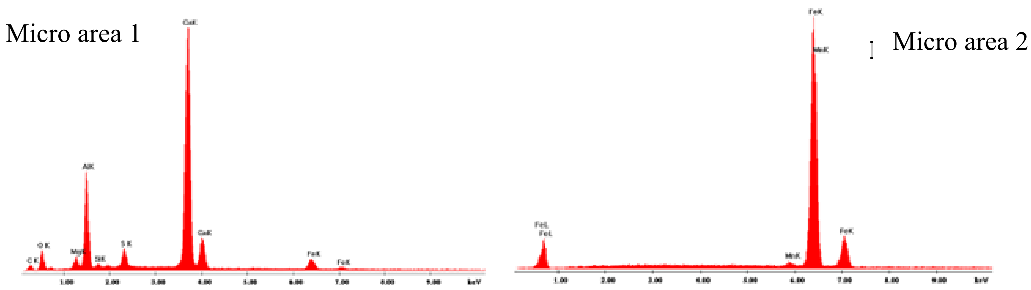

Fig. 3. Energy-Dispersive X-ray (EDAX) spectrum obtained from sample 1, micro-area 1 and micro-area 2

In contrast, the spectrum shown in Figure 3 for micro-area 2 reveals primarily Fe and Mn, consistent with the steel matrix composition.

The quantitative elemental composition for sample 1, micro-area 1 is summarized in Table 2.

Table 2

Chemical composition of non-metalic inclusion detected in sample 1, Area 1

Element	Weight (%)	Atomic weight (%)
O	26,90	44,08
Mg	3,36	3,63
Al	20,65	20,06

Si	1,07	1,00
S	3,24	2,65
Ca	41,03	26,83
Fe	3,74	1,76
Total	100	100

The analysis of Figure 4 reveals the presence of a complex inclusion composed of oxygen (O), aluminum (Al), calcium (Ca), magnesium (Mg), and sulfur (S).

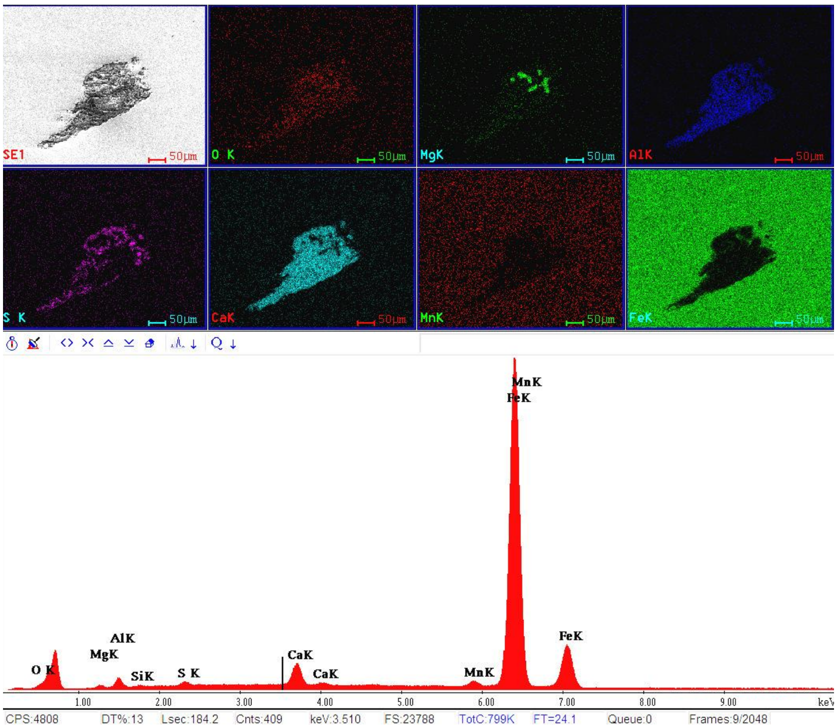

Fig. 4. Inclusion analysis – sample 1

The EDAX analysis for sample 2 is presented in Figure 5, while the corresponding energy-dispersive X-ray spectra for the analyzed micro areas are shown in Fig. 6. The quantitative elemental composition of sample 2, micro area 1 is provided in Table 3.

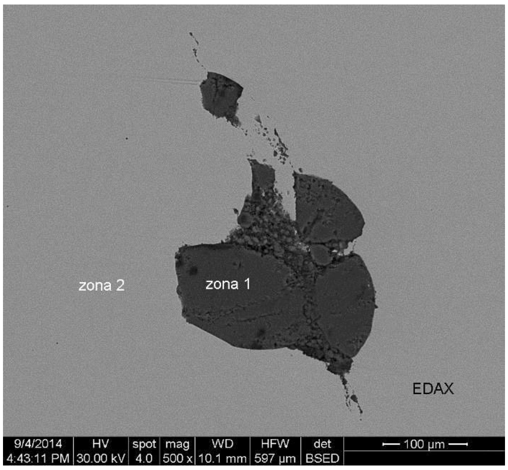

Fig. 5. EDAX Analysis, sample 2, 500x

Based on the analysis of Figure 6., in micro area 1 detected elements O, Mg, Al, S, and Ca indicate complex oxide-sulfide inclusions, more precisely formed as a result of alumina deoxidation and subsequent modification by Ca during secondary metallurgy.

Figure 6 for micro area 2 indicates the presence of Fe and Mn confirms either matrix interference or residual MnS inclusions.

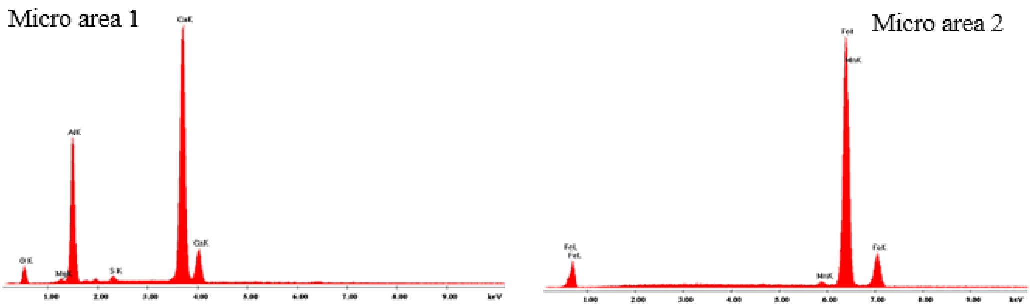

Fig. 6. Energy-Dispersive X-ray (EDAX) spectrum obtained from sample 2, micro area 1 and micro area 2

Table 3

Chemical composition of non-metalic inclusion detected in sample 2, Area 1

Element	Weight (%)	Atomic weight (%)
O	27,72	44,36
Mg	3,24	3,50
Al	21,66	20,51
Si	1,09	1,03

S	3,36	2,84
Ca	41,11	26,01
Fe	1,82	1,75
Total	100	100

Figure 7 reveals a complex inclusion composed of O, Al, Ca, and S, characteristic of calcium aluminates combined with sulfide phases, which is consistent with the expected product of SiCa wire treatment intended for Al_2O_3 spheroidization and nozzle clogging prevention.

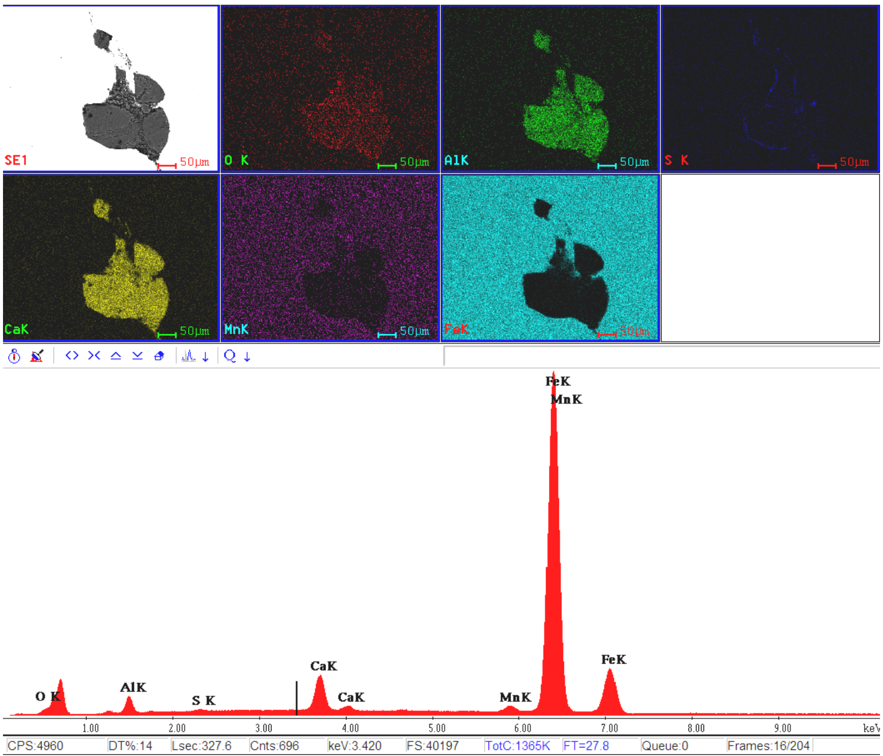

Fig. 7. Inclusion analysis – sample 2

Similarly, the EDAX results for sample 3 are shown in Figure 8, and the energy-dispersive X-ray spectra for its analyzed micro areas are presented in Figure 9. The corresponding quantitative chemical composition for sample 3, micro area 1 is summarized in Table 3.

Figure 9 in micro area 1 indicates the presence of O, Mg, Al, S, Ca, and Fe representing multi-component inclusions comprising oxides and sulfides, with Mg spinels and CaS phases being predominant. These inclusions typically originate from Al deoxidation, Mg pick-up from refractories, and Ca modification reactions.

Figure 9 micro area 2 highlights the presence of Fe and Mn, confirming contact with the steel matrix or Mn rich inclusions.

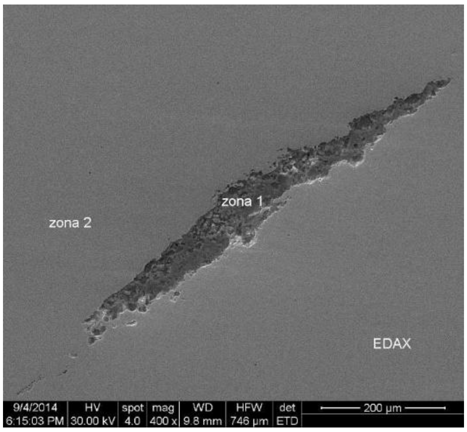

Fig. 8. EDAX Analysis, sample 3, 400x

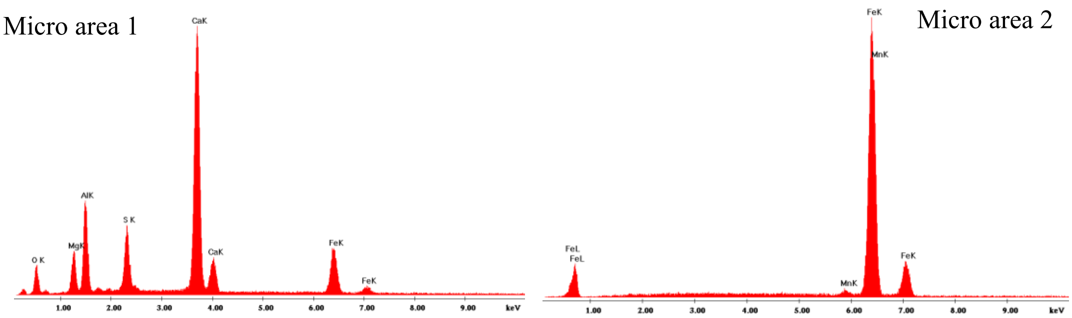

Fig. 9. Energy-Dispersive X-ray (EDAX) spectrum obtained from sample 3, micro area 1 and micro area 2

Table 4

Chemical composition of non-metalic inclusion detected in sample 3, Area 1

Element	Weight (%)	Atomic weight (%)
O	23,83	40,31
Mg	8,56	9,53
Al	16,42	16,47
S	7,70	6,50
Ca	32,10	21,67
Fe	11,39	5,52
Total	100	100

Figure 10 confirms a complex inclusion of O, Mg, Al, Ca, and S, further supporting the hypothesis of spinels and Ca-modified alumina as dominant inclusion types.

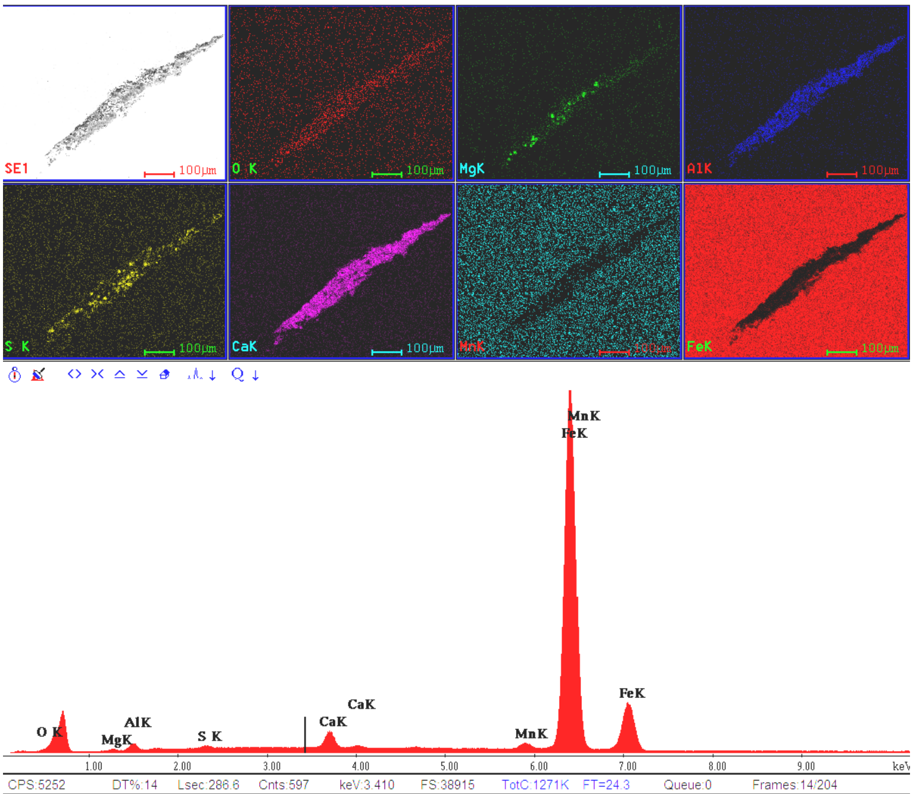

Fig. 10. Inclusion analysis – sample 3

The occurrence of Ca and Mg in inclusions reflects the applied ladle refining practices. Argon bubbling promoted emulsification at the slag–metal interface, enhancing inclusion flotation and removal. Sequential aluminum deoxidation followed by SiCa additions facilitated the transformation of angular Al_2O_3 inclusions into spherical calcium aluminates, mitigating the risk of nozzle clogging during continuous casting. Excessive SiCa was avoided to prevent CaS formation, which could compromise steel fluidity and SEN performance. Mg presence is attributed to refractory erosion, leading to $MgAl_2O_4$ spinel formation, a phenomenon commonly observed in ladle refining.

This research confirms that the combined deoxidation and calcium treatment strategy was effective in minimizing elongated alumina inclusions and promoting the formation of more globular, low-melting-point inclusion phases. Nevertheless, careful control of Ca additions remains critical to prevent detrimental CaS formation. Also combined controlled aluminum deoxidation, SiCa wire addition, and optimized argon stirring proved effective in achieving steel cleanliness, with minimized inclusion size and improved morphology. These operational practices are critical for producing high-quality semi-finished products with reduced surface defects and stable mold-level control during continuous casting.

6. Conclusions

SEM–EDS analyses revealed that the investigated inclusions are predominantly composed of O, Al, and Ca, with average concentrations ranges between 86.41–95.61 weight (%) and 89.51–96.69 atomic weight (%), with average values of 91.01 weight (%) and 93.10 atomic weight (%), respectively, confirming their classification as complex Ca–Al–O phases resulting from advanced calcium treatment. Minor alloying or process-derived elements (Mg, Si, S, Fe) were detected in limited amounts, attributed to refractory dissolution, residual deoxidation products, partial desulfurization, and steel matrix entrainment.

Minor detected elements (Mg, Si, S, Fe) represent secondary contributions from refractory erosion, residual deoxidation products, partial desulfurization, or steel matrix entrainment.

A clear inverse correlation between Fe and the combined Al + Ca content indicates efficient FeO reduction, intensified under vacuum conditions by carbon-assisted auto-deoxidation.

Sample 3 exhibits a distinct compositional deviation from samples 1 and 2, suggesting localized areas with incomplete deoxidation reactions. Fe and Mn presence in micro area 2 reflects partial entrapment of the metallic matrix or segregation during solidification.

Morphological examination showed large inclusions (>200 μm) with irregular geometries, evidencing insufficient flotation and late-stage inclusion modification prior to casting. Additional fine inclusions (<5 μm) were also observed, likely formed during deoxidation and desulfurization reactions within the melt.

R E F E R E N C E S

[1] *R. Ying, W. Weijian, Y. Wen, Z. Lifeng,* Modification of Non-metallic Inclusions in Steel by Calcium Treatment: A Review, ISIJ International, Vol. **63**, Iss.12, 2023, DOI: 10.2355/isijinternational.ISIJINT-2023-143

[2] *Y. Wen, Z. Lifeng, R. Ying, C. Wei, L. Fenggang,* Formation and Prevention of Nozzle Clogging during the Continuous Casting of Steels: A Review, ISIJ International, Vol. **64**, Iss.1, 2024, DOI: 10.2355/isijinternational.ISIJINT-2023-376

[3] *F. Xingle, Z. Lifeng, R. Ying, Y. Wen, W. Sonngjie*, The Effect of Aluminum Addition on the Evolution of Inclusions in an Aluminum-Killed Calcium-Treated Steel, Metals, Vol. **12**, Iss.2, 2022, DOI: 10.3390/met12020181

[4] *S.K. Michelic, C. Bernhard*, Significance of Nonmetallic Inclusions for the Clogging Phenomenon in Continuous Casting of Steel—a Review, Steel Research International*,* Vol. **93**, Iss.7, 2022, DOI: 10.1002/srin.202200086
[5] *L. Yuntong, H. Wenyuan, Z. Changliang, L. Jianqiu, Y. Zeyu, Z. Yuhang, Y. Jian*, Mathematical Modeling of Transient Submerged Entry Nozzle Clogging and Its Effect on Flow Field, Bubble Distribution and Interface Fluctuation in Slab Continuous Casting Mold, Metals, Vol. 14, Iss.7, 2024, DOI: 10.3390/met14070742