

DETERMINATION OF DECISIVE FACTORS INFLUENCING THE INCLUSION LANDSCAPE IN ELECTROSLAG REMELTED INGOTS

S. K. Michelic^{1*}, R. Tanzer², W. Schützenhöfer² and C. Bernhard¹

¹Chair of Metallurgy, Montanuniversität Leoben, Austria

²Böhler Edelstahl GmbH & Co KG, Kapfenberg, Austria

*Corresponding Author, contact: susanne.michelic@unileoben.ac.at

Abstract

Through Electroslag Remelting (ESR) a significant improvement of steel cleanliness can be achieved. Due to the contact of the liquid metal with the reactive slag bath at the three steel/slag interfaces not only the number and size but also the chemical composition of non-metallic inclusions changes significantly between the electrode and the remelted ingot. The present paper focuses on the modification of oxide inclusions in the hot-work tool steel X38CrMoV5-1 through Electroslag Remelting, especially concentrating on the inclusion type MgOAl_2O_3 . In general, this spinel inclusion negatively affects steel properties due to its high melting point and its undeformability at hot-forming temperatures. Next to the detailed investigation of electrode and ingot samples from the ESR process using automated SEM/EDS analysis, laboratory experiments in a Tammann Furnace were performed to study the influence of slag composition as well as of dwell time of inclusions in the liquid pool on inclusion modification. Special attention is paid to the changes in inclusion morphology which give an indication on the behaviour of inclusions during Remelting. Finally, the most decisive factors primarily influencing inclusion size and composition in the remelted ingot are summarised and evaluated.

Keywords

Remelting; inclusions; slag composition; spinel; Tammann Furnace; modification; inclusion morphology;

Introduction

The cleanliness level has emerged to an important quality criterion for a large field of high-grade steel products. In order to ensure the required properties, remelting processes have become state of the art in special steelmaking. These processes not only considerably affect the microstructure of the ingot but also significantly control and influence the content of non-metallic inclusions (NMI) in the steel matrix. Among the remelting processes is Electroslag Remelting (ESR), where the determining refining effect is achieved through the contact between the liquid metal and a reactive slag bath.

According to literature [1], principally three potential sources for non-metallic inclusions exist:

- First, inclusions may be a relic from the electrode;
- Secondly, oxygen or oxygen-affine elements dissolved in the electrode may react with other elements in the liquid melt;
- Finally, reactions between steel and slag during remelting may form or modify inclusions.

In the ESR process three steel-slag interfaces exist, each of them offering different conditions for reactions and interactions between metal, slag and non-metallic inclusions. Although reactions theoretically can take place at each of the three possible reaction sites, the electrode/slag interface is supposed to be the most efficient for the removal of

non-metallic inclusions by a couple of researchers [2-7] due to the comparably high temperatures and the increased residence time of inclusions at this site.

Regarding the question whether inclusions from the electrode may survive until the remelted ingot no consistent opinion is found in literature. Although the majority of authors [1,4] suppose that most inclusions present in the final ingot have nucleated and grown in the mushy zone, the problem of getting a reliable answer to this question is highlighted by Liddle [8]. Next to the inclusions in the electrode, slag composition and deoxidation practice are seen to have a significant impact on the final inclusion landscape in the remelted ingot [1,3,9].

Within the present work, the modification of non-metallic inclusions through Electroslag Remelting is studied, especially focusing on the spinel inclusion type MgOAl_2O_3 . For this purpose electrode and ingot samples from the ESR process are compared. Additionally laboratory experiments in a Tammann Furnace are carried out in order to examine the influence of slag composition and dwell time of inclusions in the liquid pool on the final inclusion landscape. Special attention is paid on the changes in inclusion morphology, which may give an important indication on inclusion origin and the decisive factors influencing the overall removal rate during remelting.

Experimental Procedure

A flow chart of the used experimental procedure is illustrated in Figure 1. For all experiments the hot-work tool steel X38CrMoV5-1 was used (basic composition given in Table 1). In order to investigate the influence of the electrode composition on the inclusion landscape in the final ingot two different initial electrode conditions have been examined. These two electrodes had the same basic composition but were treated differently in the melt shop at Böhler Edelstahl GmbH & Co KG, resulting in

varying contents of Al, Mg and O. A comparison between the two electrode conditions is given in Table 2.

These two electrodes were remelted in the special melt shop at Böhler Edelstahl GmbH & Co KG using a standard process slag composition, consisting of approximately equal percentages of CaO, Al₂O₃ and CaF₂ with slight additions of MgO and SiO₂. In a second step samples of the residual electrodes were taken for laboratory tests in the Tammann Furnace.

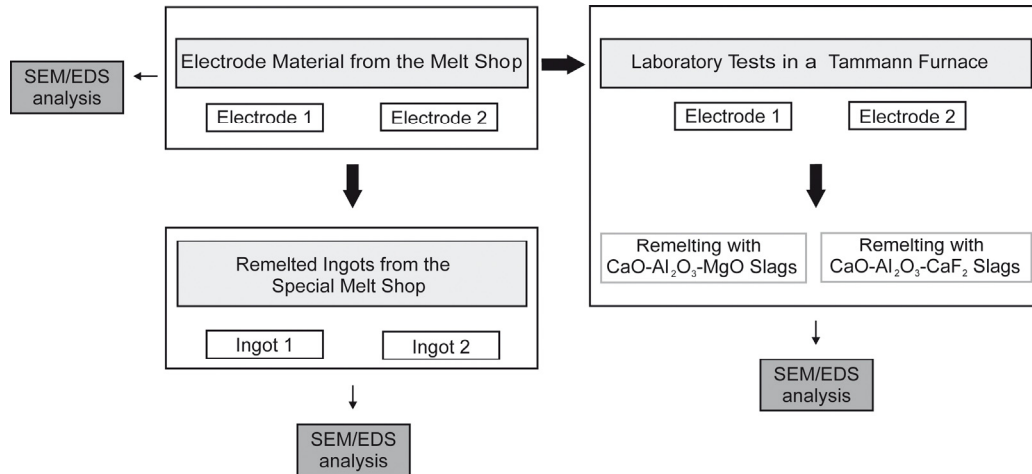


Figure 1: Flow Chart of Experimental Procedure.

wt.-% C	wt.-% Cr	wt.-% Mn	wt.-% Si	wt.-% V	wt.-% Mo
0.38	5	0.4	1.1	0.4	1.3

Table 1: Basic composition of investigated steel grade.

	wt.-%Al	wt.-% Mg	wt.-% Ca	wt.-% O
E 1	↑	↓	~	↓
E 2	↓	↑	~	↑

Table 2: Comparison of Al-, Mg-, Ca- and O-contents in the two initial electrode conditions.

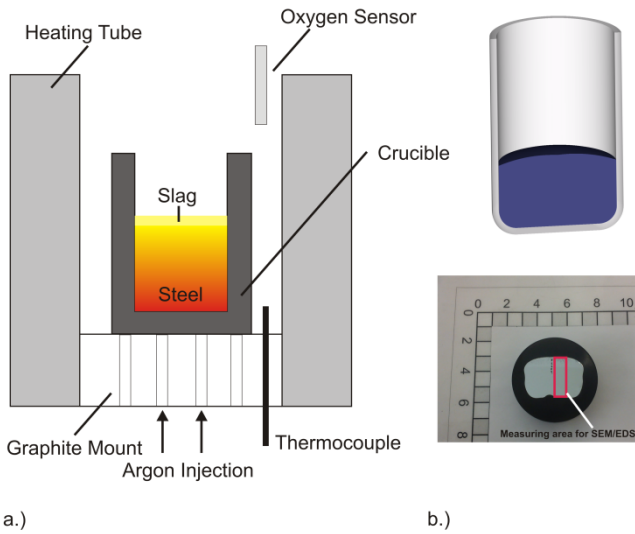
The experimental set-up of the high-temperature resistance furnace is schematically shown in Figure 2a. Further details are given in [10]. All experiments were carried out under Ar-atmosphere (Argon 5.0), using a sample weight of 100 g steel. The samples were heated up to 1600 °C; this temperature was held for 10, 20 or 30 min. The slag always covered the whole steel bath surface (the slag mass equals appr. 5 % of the steel mass).

Different slag systems have been examined, especially concentrating on the influence of the CaO/Al₂O₃ ratio as well as the CaF₂-content of the slag. Table 3 summarises the used slag compositions with continuously decreasing CaO/Al₂O₃ ratio: Slags A.1-A.3 do not contain any CaF₂, slags B.1-B.3 offer a CaF₂-content comparable to the standard ESR process slag. Regarding the CaO/Al₂O₃ ratio, slags A.1 and B.1 match the process slag composition the closest. In order to choose the slag composition for the experiments accurately, thermodynamic calculations using the commercial software FactSage 6.1 were performed also examining the liquid slag projection of the applied systems as a function of temperature. All used slag compositions are fully liquid at 1700 °C. The calculation results for the slag system CaO-Al₂O₃-CaF₂ are summarized in [11].

Next to slag composition the duration time at 1600 °C during the experiment was varied between 10, 20 and 30 min in order to simulate the influence of the

dwel time of inclusions in the liquid pool on the final inclusion landscape. For all experiments with varying slag composition a duration time of 10 min was applied. After the experiments the steel samples were prepared metallographically (see Figure 2b).

Manual and automated SEM/EDS analyses were used for inclusion characterisation in electrode and remelted ingot samples as well as in all samples from laboratory experiments.



	CaO/Al ₂ O ₃ ratio	wt.-% CaF ₂
Slag A.1*	1	0
Slag A.2*	0.5	0
Slag A.3*	0.2	0
Slag B.1*	1	30-35
Slag B.2*	0.5	30-35
Slag B.3*	0.2	30-35

* slight additions of MgO and SiO₂ each

Figure 2: a.) Tammann Furnace Design and b.) Sample position and metallographic sample for automated SEM/EDS analysis.

Table 3: Slag compositions used for laboratory experiments.

Comparison of Inclusion Landscape in the Electrodes and the Remelted Ingots

Figure 3 displays the composition of NMIs in the ternary system CaO-Al₂O₃-MgO in the two electrodes before remelting. While in electrode 1 the predominant inclusion types are Al₂O₃ and MgO, in electrode 2 a large amount of inclusions within the binary system MgO-Al₂O₃ was detected. Substantial differences are found when comparing the inclusion landscape in the remelted ingots with that of the initial electrodes. The predominant

inclusion type in both ingots is MgOAl₂O₃. The composition of NMIs within the ternary system CaO-Al₂O₃-MgO in the ingots is given in Figures 4 and 5. For each ingot the results are exemplified for two different sample positions. It is obvious that the composition of NMIs in the two remelted ingots is relatively similar, although the original states before remelting were very different.

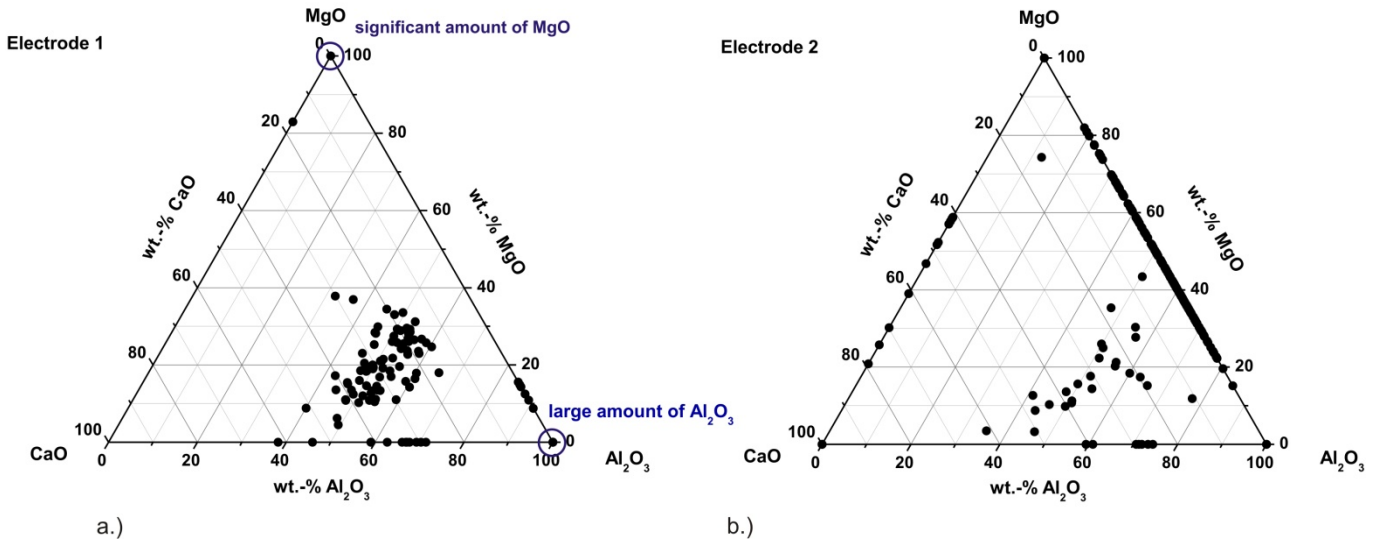


Figure 3: Initial compositions of NMIs in the ternary system CaO-Al₂O₃-MgO in the two electrodes.

In both ingots, most of the inclusions are situated in the binary system MgO-Al₂O₃, shifted to slightly higher contents of MgO in the case of ingot 2. Differences between the two ingots are observed concerning inclusions within the binary system CaO-Al₂O₃; the detected percentage of these inclusions is raised in ingot 1.

Comparing the composition of inclusions between the bottom area of the ingot and the medium ingot height a significant movement to higher Al₂O₃-contents with increasing ingot height is observed for both ingots.

This can be the result of a slightly increasing Al₂O₃-content of the slag over the process time. Besides differences in chemical composition between the two ingot sample positions, also an increase of the mean Equivalent Circle Diameter (ECD) of MgOAl₂O₃ with increasing ingot height is observed.

The performed thermodynamic calculations also show a good concordance with the practical results. As described in [11], the predicted predominant inclusion type in both remelted ingots is MgOAl₂O₃.

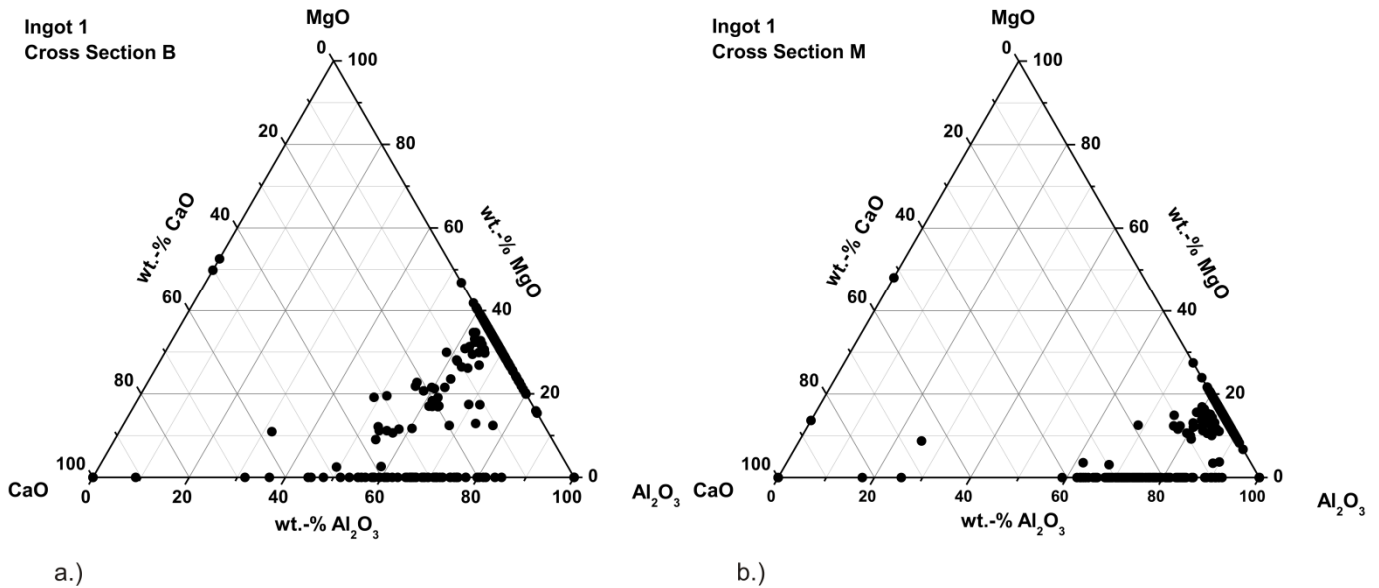


Figure 4: Compositions of NMIs in the ternary system CaO-Al₂O₃-MgO in the remelted ingot 1, for two different positions of ingot height (B=Bottom area, M= Medium ingot height).

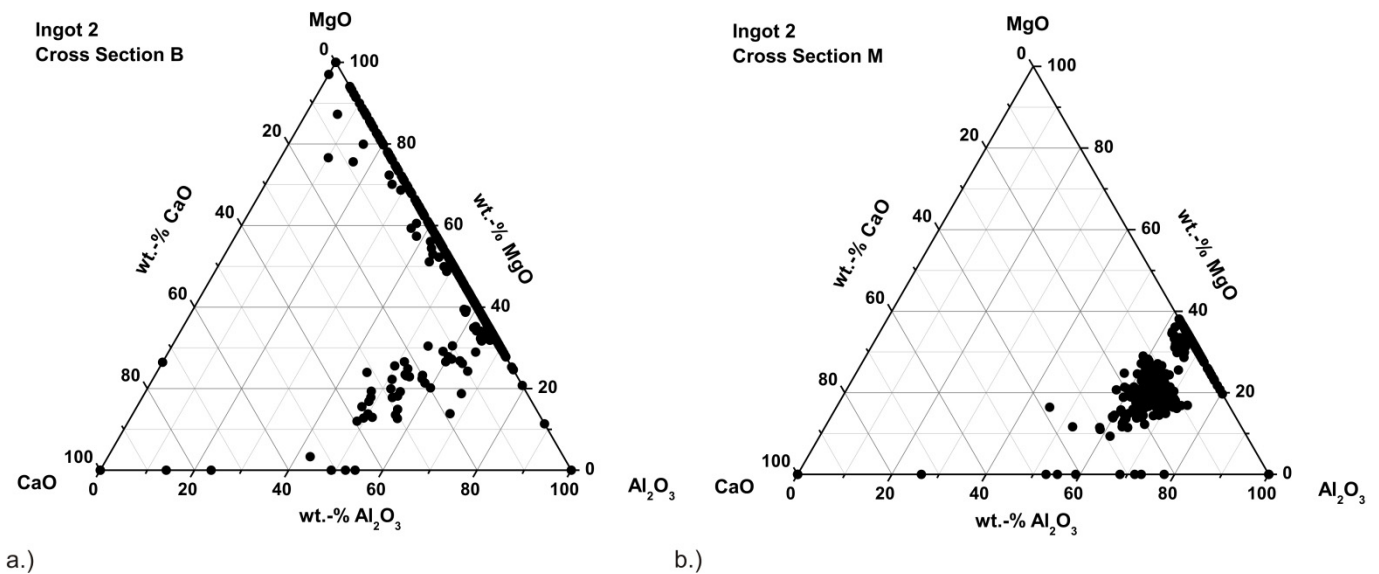


Figure 5: Compositions of NMIs in the ternary system CaO-Al₂O₃-MgO in the remelted ingot 2, for two different positions of ingot height (B=Bottom area, M= Medium ingot height).

Results of Laboratory Experiments

In the following the results of laboratory experiments are described and compared to the results of the remelted ingots from the ESR process. Principally a decrease of the overall inclusion content is achieved through remelting in all samples. Since the reaction between metal and slag is decisive for inclusion modification, the influence of different slag compositions on the change of inclusion composition is analysed. Additionally, the effect of longer dwell time in the liquid metal pool on inclusion properties is discussed.

Influence of Slag Composition

Figures 6 and 7 exemplarily show the results for remelting samples of electrode 1 and 2 with different slag compositions (see Table 3). For both cases, slags A (without CaF_2) and B, the $\text{CaO}/\text{Al}_2\text{O}_3$ ratio has been decreased continuously from slag 1 to 3. The $\text{CaO}/\text{Al}_2\text{O}_3$ ratio of slag A.1 and B.1 represents the ESR process conditions the closest. Summing up, the influence of slag composition on the final inclusion landscape can be summarised as follows:

- Remelting experiments with both slag systems principally show a similar trend as far as the change of inclusion composition within the system $\text{CaO}-\text{Al}_2\text{O}_3-\text{MgO}$ is concerned. Consequently CaF_2 seems to have only a minor influence on the change of inclusion composition through remelting compared to Al_2O_3 .
- An increasing Al_2O_3 -content in the slag causes a remarkable shift of the inclusion composition towards the Al_2O_3 -rich corner. This result also affirms the change of inclusion composition in the ESR process over the ingot height (see Figures 4 and 5), since there is also a change of the Al_2O_3 -content over the process time.
- Moreover an increase of the mean ECD of MgOAl_2O_3 is observed with increasing Al_2O_3 -content in the slag. Although the mean ECD of MgOAl_2O_3 also increases with ingot height in the ESR process, here also the longer dwell time in the pool has to be taken into account.

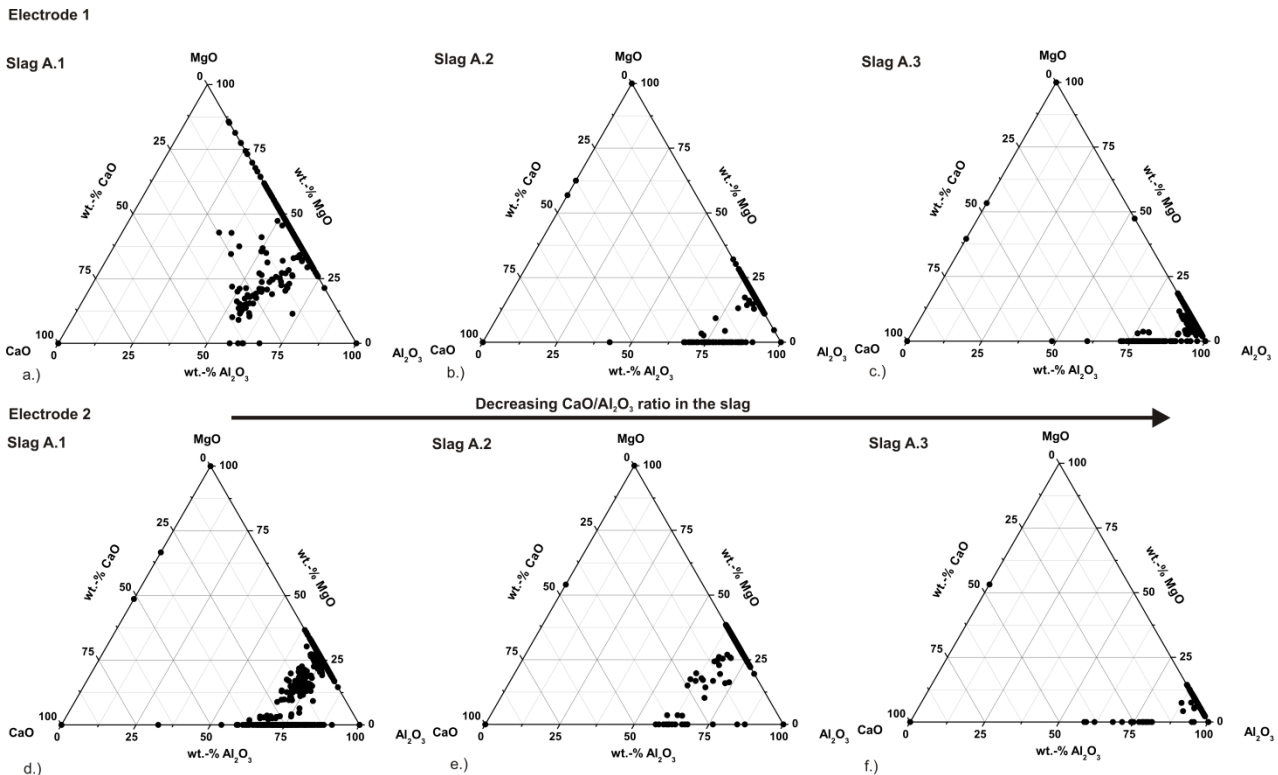
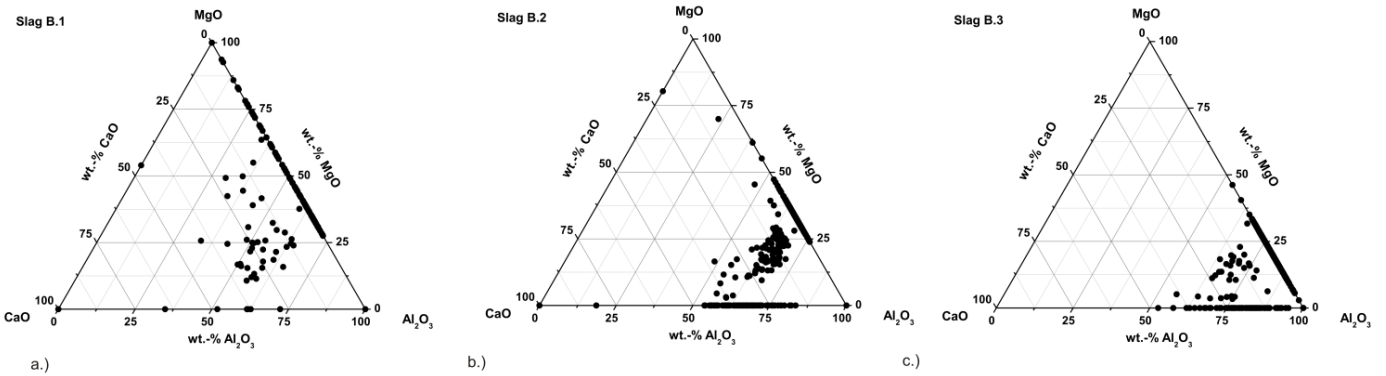


Figure 6: Compositions of NMIs in the ternary system $\text{CaO}-\text{Al}_2\text{O}_3-\text{MgO}$ in the remelted samples of laboratory experiments for varying slag compositions Slag A.1-A.3.

Electrode 1



Electrode 2

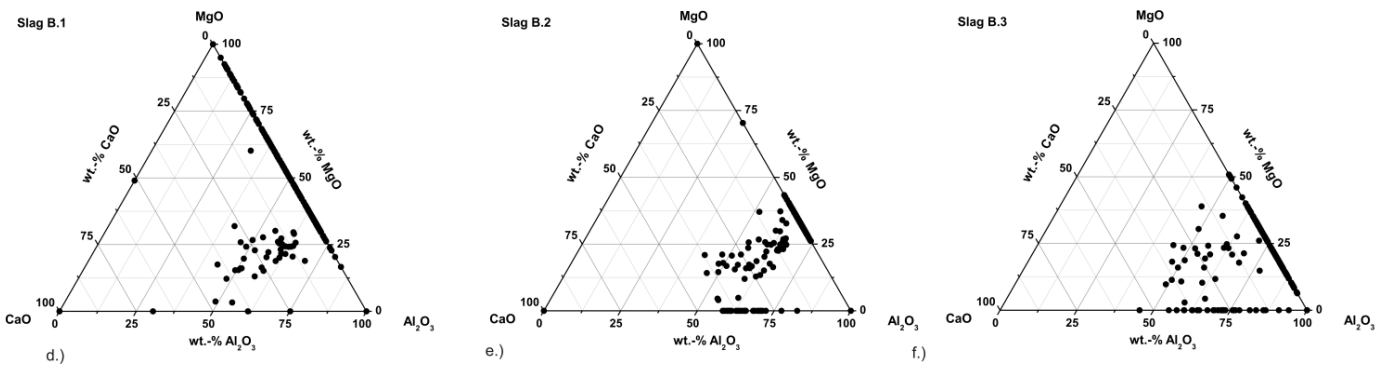


Figure 7: Compositions of NMIs in the ternary system CaO-Al₂O₃-MgO in the remelted samples of laboratory experiments for varying slag compositions Slag B.1-B.3.

Influence of Dwell time of Inclusions in the Liquid Pool

Figure 8 illustrates the effect of the duration time of the liquid melt at 1600 °C on the mean detected oxide inclusion diameter. The duration time has been varied between 10 and 30 min for both electrode conditions using slag A.1 and slag B.1 for all experiments. The main findings are summarised as follows:

- A significant increase of the mean ECD of oxides with longer duration time is observed, especially for samples of electrode 2, most effectively between 20 and 30 min.
- The mean ECD of oxides after remelting samples of electrode 2 with slag B.1 is smaller than for samples remelted with slag A.1. This may also be related to the more advantageous separation using CaF₂-slags.
- For samples of electrode 1 also a slight increase of the mean ECD is observed with longer duration time, but not as distinct as for electrode 2.

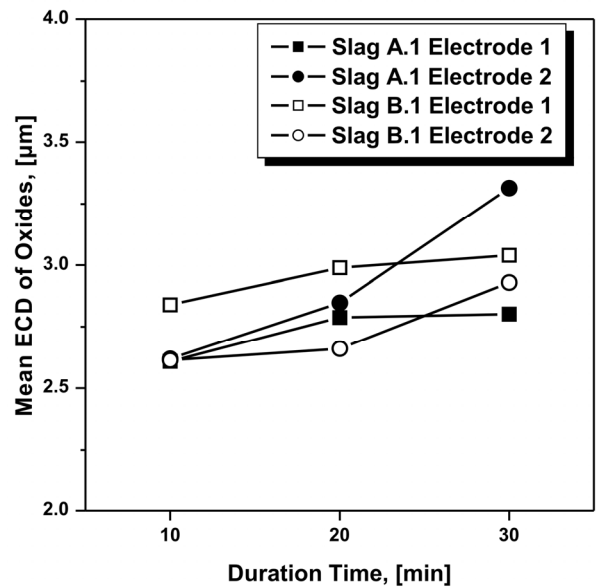


Figure 8: Relationship between the duration time of inclusions in the liquid state and the mean oxide inclusion diameter.

Significance of Inclusion Morphology

Despite different initial inclusion compositions in electrode 1 and 2, as shown beforehand the final inclusion composition within the system $\text{CaO-Al}_2\text{O}_3\text{-MgO}$ in the remelted ingots as well as the laboratory samples was similar. However, large differences concerning the inclusion morphology can be determined. While in the initial electrode samples hardly any multiphase inclusions were observed, the majority of inclusions in remelted samples (ingots as well as samples from laboratory tests) consist of two or more phases. In dependence of the initial inclusion landscape in the electrodes, the multiphase inclusions in remelted samples show a different phase appearance.

In principle, as illustrated in Figure 9 three main types of basic inclusions can be differentiated in remelted samples:

- Type 1 consists of two or more oxide phases which can clearly be separated from each other, where mostly an MgO core is surrounded by an Al-rich layer.
- Type 2 is characterized by a single phase oxide, but is surrounded by a $(\text{Ti,V})(\text{C,N})$. Thus, MgOAl_2O_3 is found to act as heterogeneous nuclei for $(\text{Ti,V})(\text{C,N})$.
- Type 3 describes a classical single phase oxide spinel inclusion.

Different Spinel Inclusions in the Remelted Samples

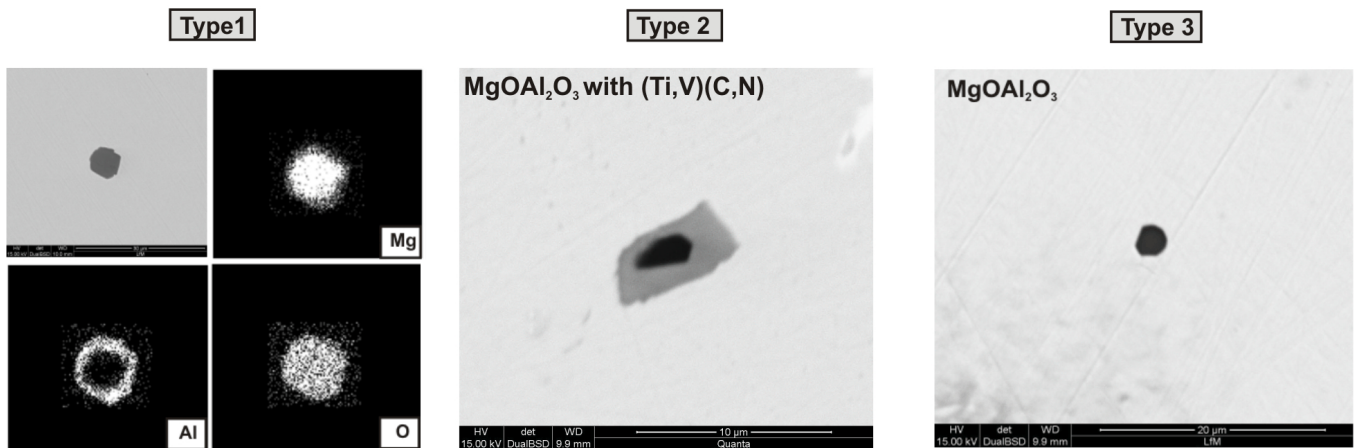


Figure 9: Different types of basic inclusions found in remelted samples.

In ingot 2 as well as in remelted laboratory samples of electrode 2 mainly inclusions of type 2 were detected, against what in remelted samples of electrode 1 and ingot 1 primarily inclusions of type 1 and type 3 were found. Moreover, accompanying kinetic calculations showed that the dissolution time of MgO and MgOAl_2O_3 in this steel composition is significantly longer than that for Al_2O_3 . Thus, MgOAl_2O_3 appears to be much more stable than pure Al_2O_3 under the defined conditions.

Based on the obtained results, it is assumed that these different morphologies also give an indication on the reaction mechanisms in ESR for the different inclusion types and a possible answer to the question, whether the inclusion in the ingot was in contact with the slag or not and therefore may be a relic from the electrode or not:

- Inclusions of type 1 and 2 are supposed to be relics from the electrode, which were not in

contact with the slag and consequently had enough time to be modified in the liquid pool.

- In contrast, type 3 seems to have nucleated in the liquid pool.

Further examples of SEM-images and corresponding mappings of inclusions detected in ingot 1 and 2 as well as in samples of laboratory tests can be found in [11].

While a change in slag composition within the investigated limits does only lead to an overall increase of the mean inclusion diameter and does not modify the inclusion morphology essentially, a longer dwell time of inclusions in the liquid metal significantly influences inclusion morphology. Based on the inclusion types shown in Figure 9 a longer dwell time favors the nucleation of additional phase on the basic inclusion. Figures 10 and 11 exemplarily display SEM-mappings of inclusions in remelted samples of electrode 1 and 2. In the case of electrode 1 (see

Figure 10), an $MgOAl_2O_3$ (basic inclusion type 3 in Figure 9) is surrounded by a $CaOAl_2O_3$ -layer. Additionally a ZrO_2 -layer resulting from reaction with the crucible material in laboratory test is formed. In the case of electrode 2 –as already shown before –

the $MgOAl_2O_3$ acts as heterogeneous nuclei for $(Ti,V)(C,N)$ (basic inclusion type 2 in Figure 9) and the longer the duration time the more likely also a Molybdenum-rich phase forms.

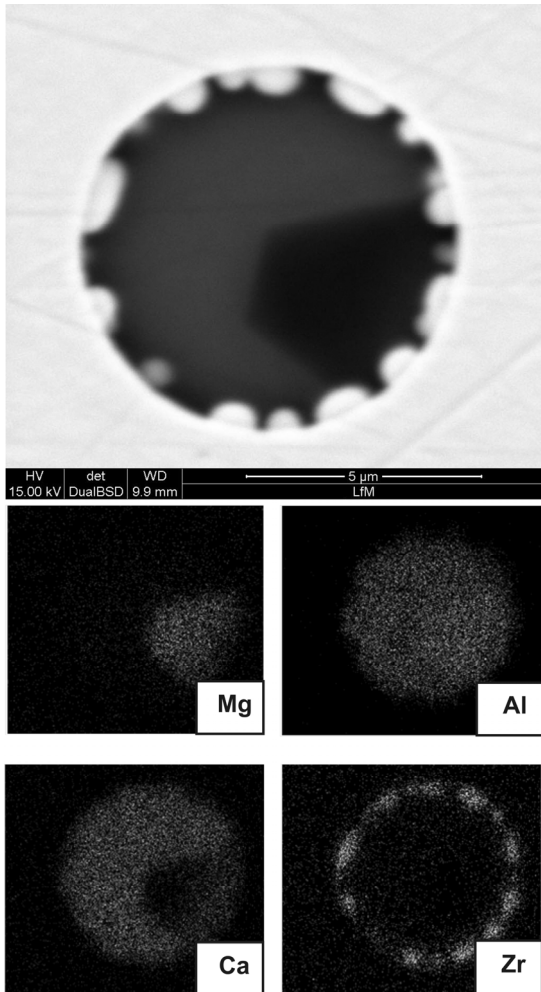


Figure 10: SEM-mapping of a typical inclusion in a sample of electrode 1 remelted with slag B.1 and a duration time of 20 min; exemplifying the modification of a basic inclusion of type 3 with longer dwell time in the liquid metal.

The influence of slag composition and dwell of inclusions in the liquid pool on inclusion morphology is schematically illustrated and summarised in Figure 12 considering the three main basic inclusion types found in the remelted samples (illustrated in Figure 9).

Both – increasing Al_2O_3 -content in the slag as well as longer dwell time in the liquid pool – lead to an increase of the medium inclusion diameter. Especially longer dwell time in the liquid pool favours the heterogeneous nucleation of carbides and nitrides or the formation of a $CaO-Al_2O_3$ -layer. This effect is especially observed on the basis of $MgOAl_2O_3$

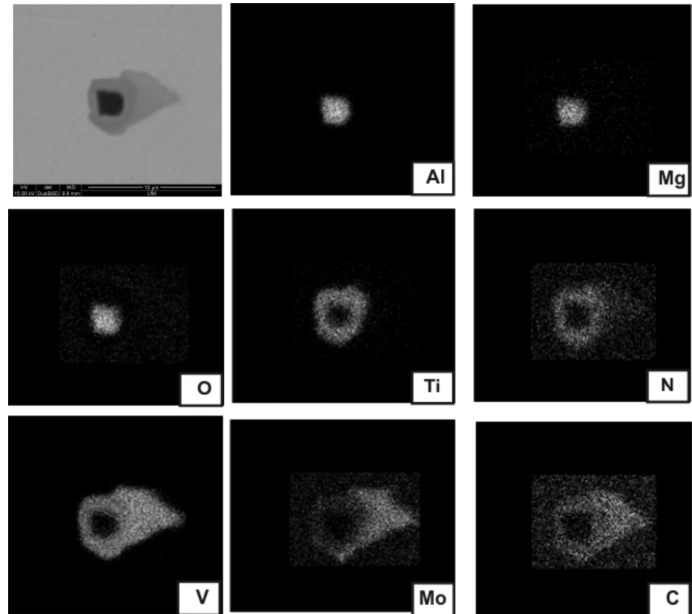


Figure 11: SEM-mapping of an inclusion in a sample of electrode 2 remelted with slag A.1 and a duration time of 30 min; exemplifying the modification of a basic inclusion of type 2 with longer dwell time in the liquid metal.

inclusions. In these cases, the $MgOAl_2O_3$ core remains unchanged. Pure Al_2O_3 inclusions acting as heterogeneous nuclei for carbides or nitrides were not found in the investigated samples.

These findings are also in good agreement with the change of the mean oxide ECD with longer duration time given in Figure 8. The more significant increase of ECD for samples of electrode 2 can be explained by the fact that $MgOAl_2O_3$ is the predominant inclusion type in electrode 2 and the nucleation of carbides and nitrides can remarkably influence the inclusion size.

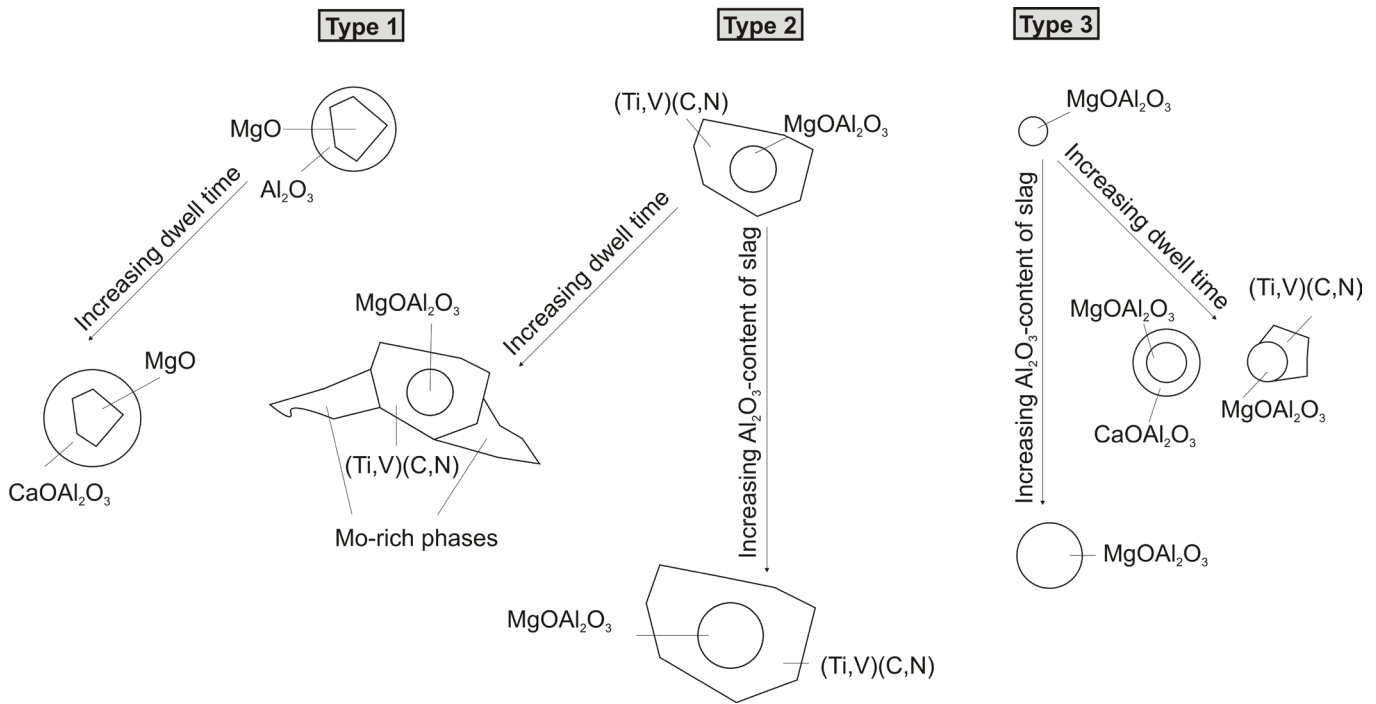


Figure 12: Schematic illustration of the influence of slag composition and dwell time of inclusions in the liquid pool on inclusion morphology in the remelted samples

Summary and Conclusion

The modification of oxides in the steel grade X38CrMoV5-1 through Electroslag Remelting has been investigated in detail using laboratory tests in a Tammann Furnace. Changes in inclusion size, composition and morphology due to steel/slag interactions are described. For this purpose the slag composition and the duration time has been varied. In a second step possible parallels to the ESR process were drawn and the decisive influencing parameters on inclusion modification were deduced. Summing up, the following main conclusions can be drawn on the basis of laboratory experiments:

- Principally a good agreement between samples from the ESR process and laboratory tests is achieved for the examined parameters.
- Regarding the used slag composition, mainly Al_2O_3 was found to essentially influence inclusion composition and inclusion size. In contrast, CaF_2 does not seem to considerably affect inclusion composition, but has an important impact on physical slag properties and therefore codetermine the removal rate of inclusions.
- Longer experimental duration times cause longer dwell time of inclusions in the liquid metal. On the one hand, this increases time for inclusion modification and especially MgOAl_2O_3 was found to act as heterogeneous nuclei for carbides and nitrides. On the other hand, longer dwell time also enhances the possibility of inclusion separation through flotation. Consequently, an agreement of a most adequate dwell time has to be defined weighing the involved advantages and disadvantages.
- The initial inclusion composition in the electrode highly influences the final inclusion size and morphology in the remelted ingot. Regarding the overall removal rate and the final inclusion content, the inclusion composition in the electrode is seen to be more important than the initial inclusion number or size.
- The morphology of inclusions in remelted samples is seen to be a decisive indicator for the understanding of inclusion behaviour during remelting.

Abbreviations

ECD	Equivalent Circle Diameter
ESR	Electroslag Remelting
NMI	Non-metallic inclusion

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