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Utilizing inclusion data in characterization of oxide-sulfide stringers in hot-rolled plates

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The targets of this study were to determine the effect of vertical location and the composition of inclusions to the occurrence of oxide–sulfide stringers, and to determine the calcium aluminate phases most prone to form stringers during hot rolling of aluminium killed, calcium treated steel. The phases present in the inclusions have a significant effect on the deformation of inclusions during hot rolling, and consequently, on mechanical properties of the steel. A MATLAB script is utilized to identify and locate detrimental stringers from the hot rolled plates. Inclusion analysis data gathered with a scanning electron microscope and exported from IncaFeature software is analyzed. The following properties are presented for each stringer: the number of inclusions and length of stringer, phase fractions and compositions, and the composition of the unfragmented inclusion before hot rolling. According to the results, the longest stringers have total lengths over 200 µm, with almost 20 inclusions. The overall composition of the longest stringers is between C12A7 and C3A calcium aluminates with minor MgO contents. The diameters of the unfragmented inclusions in the slabs, forming stringers during hot rolling, were estimated to be around 20 µm for the longest stringers. From the dataset, plenty of CaO–CaS stringers were also characterized, obviously a result of excess calcium treatment.

1. Introduction

Oxide-sulfide stringers are considered harmful in hot rolled steel plates, similarly to elongated manganese sulfides [1]. In aluminum-killed and calcium treated steels, the stringers are often a mixture of oxides in Al\textsubscript{2}O\textsubscript{3}-CaO-MgO system and calcium sulfides (CaS), occasionally accompanied with manganese sulfides (MnS) and titanium nitrides (TiN). The formation and characteristics of oxide-sulfide stringers is reported in recent studies [1, 2]. The phases present in the inclusions have a marked effect on the deformation of inclusions during hot rolling, and consequently, on mechanical properties of the steel. Still, no consensus exists on the deformation tendency of various calcium aluminate phases, for example.

The developed method is used to locate and characterize oxide–sulfide stringers by using Excel spreadsheets exported from Oxford Inca Feature software. A schematic view of the formation of oxide–sulfide stringers is illustrated in figure 1. The targets of this study were to determine the effect of vertical location and the composition of inclusions to the occurrence of stringers, and to determine the calcium aluminate phases most prone to form oxide–sulfide stringers during hot rolling.
2. Materials and Inclusion Characterization Methods

In this study, 25 heats of aluminum killed, calcium treated steels produced in the SSAB Europe Raahe steel plant were investigated. Two samples were prepared from the hot rolled product of each heat, making up a total of 50 samples.

The inclusion characterization was conducted in the SSAB Europe Raahe steelworks. Inclusions were analyzed with a Jeol JSM-7000F FESEM equipped with an EDS detector. Inclusion analyses were acquired with Oxford IncaFeature software using an acceleration voltage of 15 kV. In the INCA Feature runs, inclusions larger than 1.0 µm were analyzed with a livetime of 1.0 second with EDS. The total sample area comprised of product thickness, approximately 12 mm, width being 4 mm, resulting in approximately 48 mm$^2$ of scanned area per sample.

3. MATLAB script

3.1. Stringer detection

The oxide–sulfide stringers are located by a MATLAB script that handles Excel spreadsheets exported from Inca Feature software. The sample area is analyzed by going through the whole sample area with horizontal strips, parallel to the rolling direction. The strips cover the sample width, and the vertical thickness is defined as parameter. If multiple inclusions on a strip are close enough to each other, the inclusions are identified as a stringer. In figure 2, it can be seen that the consecutively analyzed strips are overlapping in order to ensure the stringer detection. In this case, the stringer is completely discovered in the strip illustrated in figure 2c, and taken into account.

To locate stringers in the samples, various parameters are used in the MATLAB script. In Table 1, the parameters used in this case, including the ones presented in figure 2, are presented. The parameters are chosen in a way that they eliminate other than oxide-based stringers, such as manganese sulfides. In addition, the stringers must be horizontal, i.e., assumingly formed during the hot rolling of the steel.
Table 1. Applied parameters in the stringer detection script.

| Parameter                                           | Threshold value | Note                                                                 |
|-----------------------------------------------------|-----------------|----------------------------------------------------------------------|
| Number of inclusions                                | ≥ 3             | After deducting inclusions which contain TiN + MnS in total over (90 mol.%) |
| Horizontal distance between inclusions              | < 30 µm         |                                                                      |
| Average horizontal distance between inclusions      | < 20 µm         |                                                                      |
| Strip thickness                                     | 8 µm            |                                                                      |
| Strip thickness step                                | 2 µm            |                                                                      |
| At least one inclusion must contain oxides          | > 10 mol.%      |                                                                      |
| Calculated TiN + MnS phase fractions in undeformed inclusion in total | < 50 mol.%      |                                                                      |
| Stringer aspect ratio OR                            | > 8             | Eliminates clusters and inclined stringers                           |
| $R^2$ value of linear fit of inclusion coordinates AND | > 0.9           |                                                                      |
| Stringer aspect ratio                               | 2 < AR < 8      | Stringer length / Stringer breadth                                   |

3.2. Original inclusion volume estimation

In this context, “original inclusion” refers to the assumed single inclusion in the slab, which has fragmented into several smaller inclusions, i.e., a stringer during hot rolling. The size of the original inclusion corresponds to the estimated total volume of stringered inclusions. For convenience, the size of the original inclusion is presented as a diameter of a sphere with the same volume.

The inclusions are assumed ellipsoids with the major axis parallel to the rolling direction, as presented by Fuhr et al. [3]. The observed inclusion area is considered the area of a projection of an ellipsoid. Further, the aspect ratios for the observed inclusion and the approximation are assumed equal.

3.3. Original inclusion composition estimation

The inclusions are taken as a mixture of CaS, MnS, TiN and oxide (Al$_2$O$_3$-CaO-MgO), which are the most common phases in inclusions in aluminum killed, calcium treated steels [4]. The phase fractions and oxide phases composition are calculated from the elemental analysis for each inclusion. The linear set of equations to calculate the compound fractions in each inclusion is presented in equation 1, where $M_i$ is the molar mass of element or component i, $n_i$ is the amount of compound i in inclusion, $m_i$ is the mass of element i in inclusion, and $n_i \geq 0$.

$$
\begin{bmatrix}
2M_{Al} & 0 & 0 & 0 & 0 & 0 \\
0 & M_{Ca} & M_{Ca} & 0 & 0 & 0 \\
0 & 0 & 0 & M_{Mg} & 0 & 0 \\
0 & 0 & 0 & 0 & M_{Mn} & 0 \\
0 & 0 & M_{S} & 0 & M_{S} & 0 \\
0 & 0 & 0 & 0 & 0 & M_{Ti}
\end{bmatrix}
\begin{bmatrix}
\rho_{Al2O3} \\
\rho_{CaO} \\
\rho_{MgO} \\
\rho_{MnO} \\
\rho_{MnS} \\
\rho_{TiN}
\end{bmatrix}
= 
\begin{bmatrix}
m_{Al} \\
m_{Ca} \\
m_{Mg} \\
m_{Mn} \\
m_{S} \\
m_{Ti}
\end{bmatrix}
(1)

The composition of the original inclusion is the average phase composition of the stringered inclusions, weighted with the volumes of the stringered inclusions. The oxide phases in each inclusion is calculated from the total composition, according to the lever rule. In this study, the focus is on various calcium aluminates, with compositions of CA, C12A7, and C3A.

4. Results and Discussion

The following properties are stored for each stringer: the number of inclusions and length of stringer, phase fractions and compositions, and the composition of the unfragmented inclusion before hot rolling.
In figure 3, an example of observed stringered inclusion and its geometrical visualization is illustrated. The calculated phase fractions and oxide compositions for each inclusion are also presented.

![Figure 3](image)

**Figure 3.** Stringered inclusion observed in hot-rolled product sample (left), phase fractions and oxide compositions (top) and geometrical visualization of inclusions (bottom right).

Most of the long stringers, with total lengths over 100 µm, locate on the upper half of the rolled product, as seen in figure 4 (left). On the other hand, shorter stringers are more evenly dispersed throughout the thickness of the product. The longest stringers have total lengths of over 200 µm in the studied samples. The longest stringers comprised of 19 inclusions (figure 4, right). On the lower side of the hot rolled product, only one stringer including more than ten inclusions is observed.

![Figure 4](image)

**Figure 4.** Length of stringers as a function of stringer location.

The size of original inclusions was estimated and shown in figure 5. The longest stringers have formed from inclusions with sizes of 10–17 µm, assuming they were spherical inclusions in the slab before hot rolling. The original inclusion sizes calculated here are in accord with the values reported by other authors [1, 3].
Here, the correlation between fractions of calcium aluminates and calcium oxide in the original inclusions and the thickness location of stringers on the hot rolled product is assessed. Stringers containing CA calcium aluminate phases are often under 100 µm, illustrated in figure 6 (left). However, stringers with lengths over 150 µm contain marked amounts C12A7 (figure 6, right) or C3A phases (figure 7, left). Typically, the longest stringers contain also minor amounts of MgO, but negligible amounts of CaS phases. Plenty of CaO–CaS stringers were observed, obviously formed due to excess calcium treatment. The CaO-containing stringers are recurrently shorter than 50 µm, as seen in figure 7 (right).

**Figure 5.** Estimated diameter of unfragmented inclusions.

**Figure 6.** Calculated fractions of CA and C12A7 of original inclusion as a function of stringer length.
5. Summary

Utilizing the MATLAB script, the number and properties of oxide–sulfide stringers are found. Excel spreadsheets exported from Inca Feature software act as input files without further formatting. The stringers are rapidly located from the dataset, providing additional information on the inclusion data in hot-rolled product samples. With a desktop computer with Intel i7 processor running at 3.40 GHz, a typical time to analyze the stringers in on sample takes less than two minutes.

According to the results, the longest stringers have total lengths over 200 µm, with almost 20 inclusions. The typical composition of long stringers is C12A7–C3A calcium aluminates with minor MgO contents, but negligible amount of CaS. The diameters of the unfragmented inclusions in the slabs, forming stringers during hot rolling, were estimated to be around 20 µm for the longest stringers. A large number of relatively short CaO–CaS stringers were observed, an indication of excess calcium treatment.

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