APPLICATION NOTE

# Characterization of complex inclusions in steel using ColorSEM Technology

## Large inclusion analysis

Steelmaking is a highly oxidizing process, through either the integrated basic oxygen process or the electric arc furnace scrap melting route. Deoxidation by metallic Mn, Si or Al removes much of the dissolved oxygen, but billions of inclusions will remain in every ton of steel. Properly identifying large (from 25 µm to 250 µm), complex inclusions in a finished product is necessary in order to create a process solution.

Elemental analysis with a scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDS) is the technique of choice for dealing with steel inclusions at the microscale. Here, we look at complex inclusions using a new EDS-based technique: Thermo Scientific™ ColorSEM™ Technology.

In Figure 1, we see a typical inclusion in steel, imaged with a SEM. Properly determining the liquid and solid phase composition will help to identify the processing conditions that formed this inclusion.

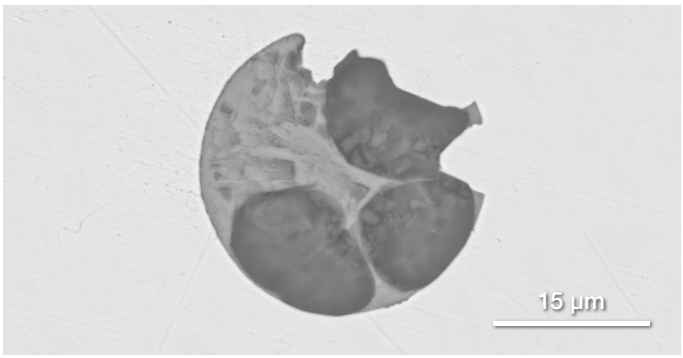


**Figure 1. Backscattered electron image.**

This image is acquired with a backscattered electron detector (BSD) that shows clear materials contrast between the steel and the inclusion, as well as contrast within the inclusion itself. While this already tells us that this is a complex inclusion with multiple phases, BSD imaging alone is not able to identify and classify the inclusion and its different phases.

Once an inclusion is identified, a typical EDS workflow would switch to the EDS computer, set up the EDS detection system, and then start to acquire data. The characteristic X-rays of the sample then allow composition analysis. This workflow could be streamlined to save time and to bring more clarity.

Here, we are using EDS integrated with the SEM user interface as part of the ColorSEM Technology implementation (Figure 2). As part of the SEM image, X-ray data is already acquired, eliminating the need either for a separate scan or to switch to a different detection system.

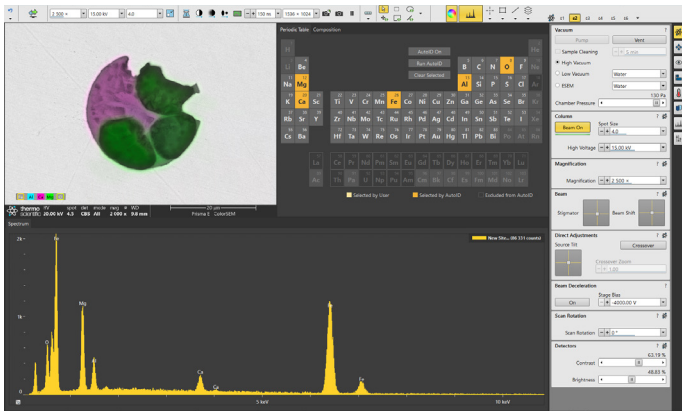

**Figure 2. User interface with a dedicated ColorSEM Technology workspace.**

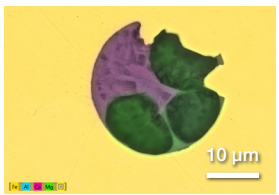


**Figure 3. ColorSEM Technology image of the inclusion shown in Figure 2 in grayscale.**

| SEM type | W gun |
|---|---|
| Acc. voltage | 20 kV |
| Beam current | 0.6 nA |
| Image resolution | 1536x1094 |
| Acquisition time | 60 s |

**Table 1. Image acquisition parameters.**

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In Figure 3, the ColorSEM image of the inclusion is shown. Elements are automatically identified by the AutoID function, and, because of the elemental coloring, additional contrast is shown. The elemental maps are the result of a quantification routine that considers such things as peak overlaps to avoid incorrect element labeling.

Each element can be separately switched on or off to quickly assess the distribution of single elements. This saves time, because a single frame acquisition allows you to save an entire set of images.

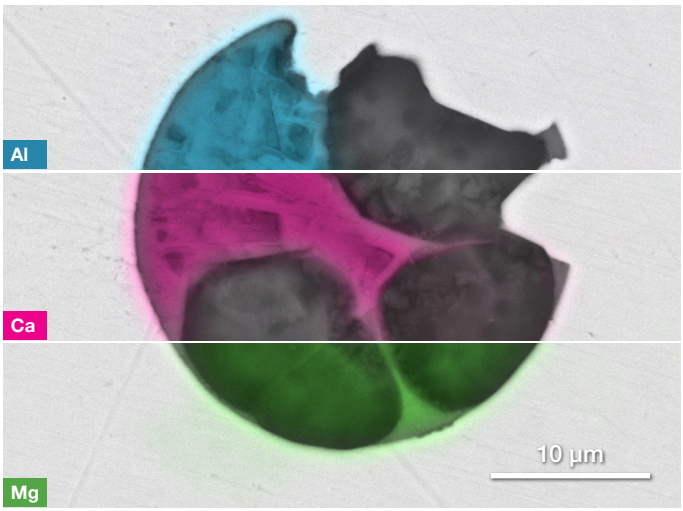



**Figure 4. ColorSEM Technology images of the distribution of aluminum , calcium and magnesium.**

The ~25 um diameter inclusion shown in Figure 4 consists of two distinct phases.. The main globular oxide, which is light gray in the BSD image, contains primarily calcium (red) and aluminum (blue), whereas the secondary oxide phases contain magnesium. Thus a raw material containing MgO became trapped in the slag and did not dissolve (MgO-saturated slag).

For a more localized analysis, Point&ID analyses have been carried out on two different points (Figure 5), each related to one of the possible phases.

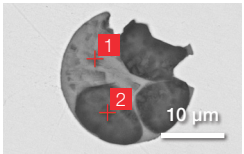



**Figure 5. Point&Id analyses.**

| Acc. voltage | 20 kV |
|---|---|
| Acquisition time | 120 s |
| Average count rate | 6,000 cps |
| Average total counts | 700,000 |

**Table 2. Point&ID parameters used for Point 1 and Point 2 analyses.**

Atomic percentages, with the related error, of the main globular oxide are presented in Table 3.

| Element | Atomic % | Atomic % error |
|---|---|---|
| O | 54.0 | 0.4 |
| Mg | 1.6 | 1.2 |
| Al | 22.4 | 0.2 |
| Si | 0.9 | 2.0 |
| S | 0.8 | 0.0 |
| Ca | 20.3 | 0.2 |

**Table 3. Point 1 quantitative analysis.**

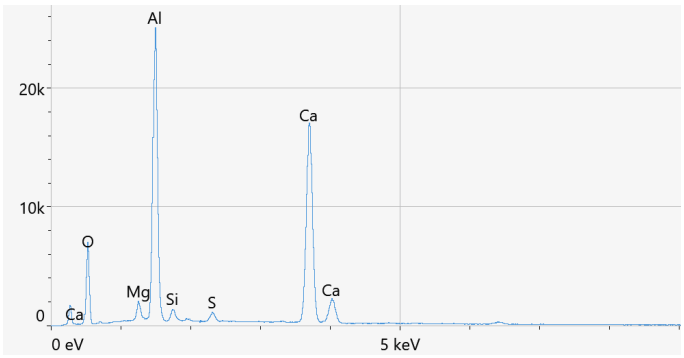



**Figure 6. Spectrum of Point 1.**

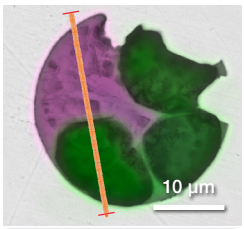
A fast data reporting function allows you to save both spectrum (Figure 6) and quantification in one click, from all the points and area analyzed. Based on the quantification extracted from Point 1, this area can be classified as a liquid calcium aluminate or $12CaO.7Al_2O_3$. Quantification obtained from Point 2, however, can be assigned to magnesium oxide (MgO).

| Element | Atomic % | Atomic % error |
|---|---|---|
| O | 50.7 | 0.3 |
| Mg | 49.3 | 0.2 |

**Table 4. Point 2 quantitative analysis.**

## Linescan characterization

In addition to EDS mapping and point analysis, a linescan can add further detail to a study. For example, the BSD image can identify most phases by grayscale, but within one phase, a change in composition may go unnoticed. The ColorSEM Technology linescan functionality allows lines to be drawn directly on the image within the user interface with no need to switch to another software program.




| Acc. voltage | 20 kV |
|---|---|
| Number of points | 200 |
| Acquisition time | 200 s |
| Average count rate | 9000 cps |
| Average total counts | ≈ 2 million |

Table 5. Linescan acquisition parameters

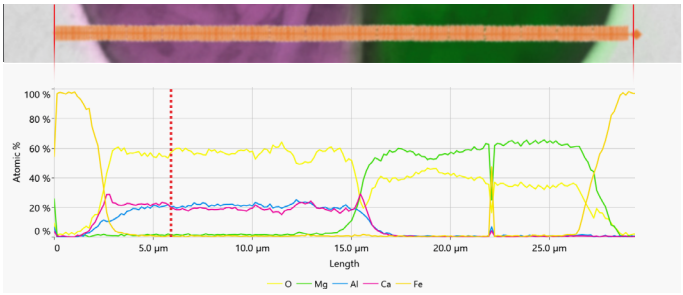



Figure 7. Linescan acquired with 200 points along the highlighted line.

A linescan of 200 points has been collected along the different phases to analyze the variations of the elements within each single area. The linescan confirms the elemental composition of the two phases and provides more details. The left part of the linescan presents a higher content of aluminum and calcium, while the magnesium is absent. Additionally, it becomes evident that the calcium content increases at the edges of the liquid calcium aluminate inclusion. The data reporting function for the linescan, apart from the elements profile, saves a spectrum and a quantification for each and every point analyzed. Atomic percentages of one of the analyzed points have been extracted from the point, indicated with a red dotted line in Figure 7.

| Element | Atomic % | Atomic % error |
|---|---|---|
| O | 54.5 | 3.2 |
| Mg | 1.6 | 8.3 |
| Al | 21.5 | 1.3 |
| Ca | 22.4 | 1.4 |

Table 6. Composition extracted from one of the points in the linescan.

The quantification in Table 6 is comparable with the results obtained from the Point&ID analysis presented previously. Nevertheless, it must be emphasized that the results in Table 6 correspond to an acquisition of 1 s. This feature, therefore, confirms the reliability of ColorSEM Technology, along with fast analysis.

## Small inclusion analysis

In many clean steels, there are very few inclusions over 100 µm long after rolling. Conversely, there will be billions of oxide and sulfide inclusions under 5 µm for every ton of steel. These small non-metallic inclusions can lead to tundish nozzle clogging or stopper rod erosion. Rapid EDS mapping of these features is needed to correlate the smaller building blocks to the oversized, agglomerated defect inclusions.

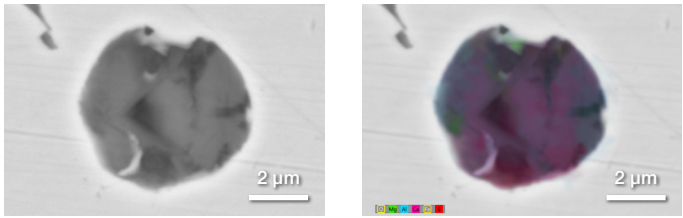



Figure 8. BSD image of a 4 µm inclusion (on the left) and the related ColorSEM Technology image (on the right).

| SEM type | W gun |
|---|---|
| Acc. voltage | 20 kV |
| Beam current | 0.6 nA |
| Image resolution | 3072x2188 |
| Acquisition time | ≈ 80 s |

Table 7. Image acquisition parameters

Both the backscattered electron image and the ColorSEM Technology image exhibit some compositional contrast that changes within more than one area inside the inclusion. Despite the information provided, the distribution of the elements is not yet fully clear. Thanks to the possibility of selecting (and subsequently deselecting) different elements, ColorSEM Technology is able to point out their variation within areas smaller than 300 nm.

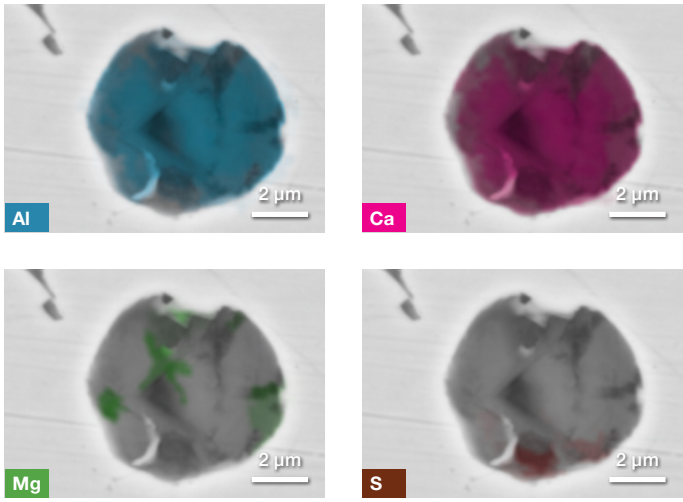



Figure 9. Distribution of elements.

The images in Figure 9 give the distribution of the involved elements, showing that the bulk oxide composition is similar to the oxide in the large inclusion described earlier. However, the presence of sulfur and magnesia in discrete spots is not readily shown by the BSD image; thus, these details might otherwise have been missed.

For a quantitative result, Points analyses were collected on three different spots (Figure 10).

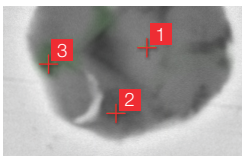



Figure 10. Poing analysis

| Acc. voltage | 20 kV |
|---|---|
| Acquisition time | 200 s |
| Average count rate | 6,000 cps |
| Average total counts | 700,000 |

Table 8. Point analysis parameters

Tables 9, 10, and 11 show the quantifications obtained, respectively, from Point 1, Point 2, and Point 3.

| Element | Atomic % | Atomic % error |
|---|---|---|
| Mg | 4.0 | 1.1 |
| Al | 45.5 | 0.2 |
| S | 3.5 | 1.0 |
| Ca | 47.0 | 0.2 |

Table 9. Point 1

Table 9 quantifies the bulk oxide composition with primarily calcium and aluminum.

| Element | Atomic % | Atomic % error |
|---|---|---|
| Mg | 2.6 | 2.2 |
| Al | 22.8 | 0.3 |
| S | 24.9 | 0.3 |
| Ca | 49.7 | 0.3 |

Table 10. Point 2

Table 10 shows irregular concentrations of sulfur that were not obvious on the BSD image. A calcium sulfide-rich phase is present here.

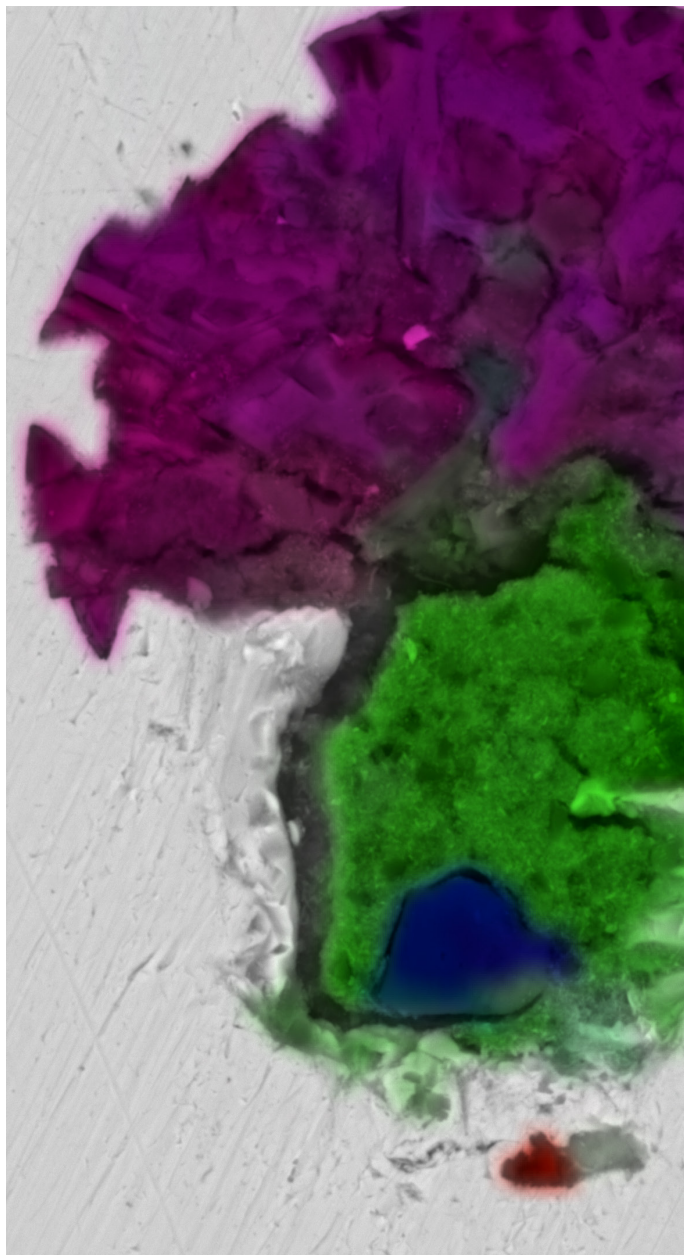
| Element | Atomic % | Atomic % error |
|---|---|---|
| Mg | 29.2 | 0.4 |
| Al | 39.5 | 0.4 |
| S | 4.8 | 0.9 |
| Ca | 26.5 | 0.4 |

Table 11. Point 3

Table 11 shows irregular concentrations of magnesium that also were not obvious on the BSD image. A magnesia-rich phase is present here.

## Conclusion

Complex oxides that arise from the steelmaking process may have several discrete phases and variations within the liquid phase. Production and quality assurance teams need to know the specific composition of each phase to determine the root cause of oversized inclusions in the final product. The "always on" EDS mapping of ColorSEM Technology makes the identification of complex oxides simpler and faster.



Find out more at **thermofisher.com/colorsem**