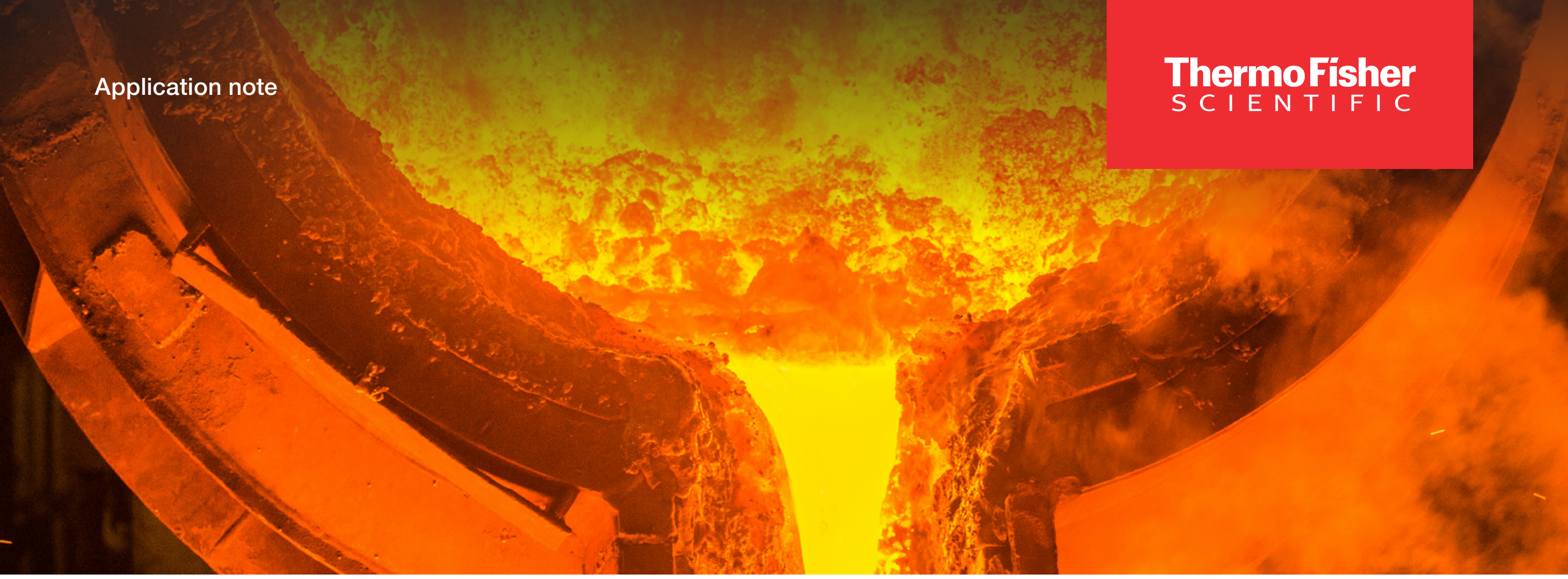

Application note

Thermo Fisher SCIENTIFIC

Inclusion analysis of complex titanium-magnesium treated steel

Melt shop alloying and refining trial evaluated with automated scanning electron microscopy (SEM)

Introduction

Low-carbon steel produced throughout the world is used for numerous applications such as automobiles, construction and, oil and gas pipelines. Many of these applications utilize aluminum to create a fine-grained microstructure due to the precipitation of nanoscale aluminum nitride that pins grain boundaries. One of the downsides to aluminum deoxidation is that it leaves behind a population of micron-sized oxides, which are prone to agglomeration and become crack initiators during bending, welding, or fatigue testing. Figure 1 shows a typical inclusion distribution for an aluminum-killed, calcium treated steel.

An alternative approach is taken in this study, which utilizes titanium, magnesium, and calcium for deoxidation. The trial was conducted through the integrated blast furnace–basic oxygen furnace route while including ladle metallurgy and RH degassing for refining before continuous slab casting.

SEM distinguishes particles from the matrix by setting a threshold on the backscattered electron image. Most steel inclusions have a lower average atomic weight than the iron matrix, showing up as dark particles in a brighter background. Energy dispersive X-ray spectroscopy (EDS) collects chemical information on each inclusion, which can then be plotted on a ternary diagram. The color represents the size, and the location represents the normalized composition for the elements represented on the corners.

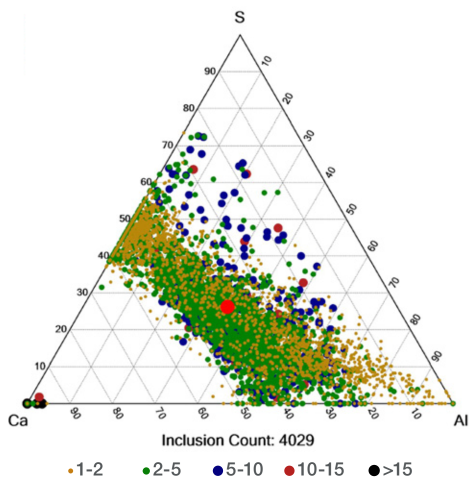

Figure 1. An inclusion population for an Al-killed, Ca-treated steel contains many calcium sulfides (left axis), calcium aluminates (center), and spinel inclusions in this 50 mm2 scan.

Element	C	Mn	Si	Ti	Mg	Ca	N	S
Wt.%	0.07	1.65	0.12	0.013	0.003	0.003	0.005	0.006

Figure 2. Bulk steel composition for the industrial trial heat.

Methods and results

Automated inclusion analysis was conducted to reveal the chemical composition of the non-metallic particles during refining. The Thermo Scientific™ Phenom ParticleX™ Steel Desktop SEM utilizes integrated EDS software and hardware that enables rapid particle detection and classification. Analysis goals were to characterize any inclusion greater than 1 µm diameter over 70 mm^2 of polished steel.

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Ladle refining began with manganese and silicon added as the primary source of deoxidation, which should yield a total oxygen content of around 50 ppm. Then the lump FeTi and wire NiMg alloys were added to the melt, further lowering the total oxygen. At this stage, a large population of titanium nitrides, magnesium oxides, and manganese/calcium sulfides were formed. A single ternary diagram cannot represent all phases here, as there are four or more unique compounds that could be represented. These diagrams show a continuous distribution of particles between two phases (either TiN + sulfides or MgO + sulfides).

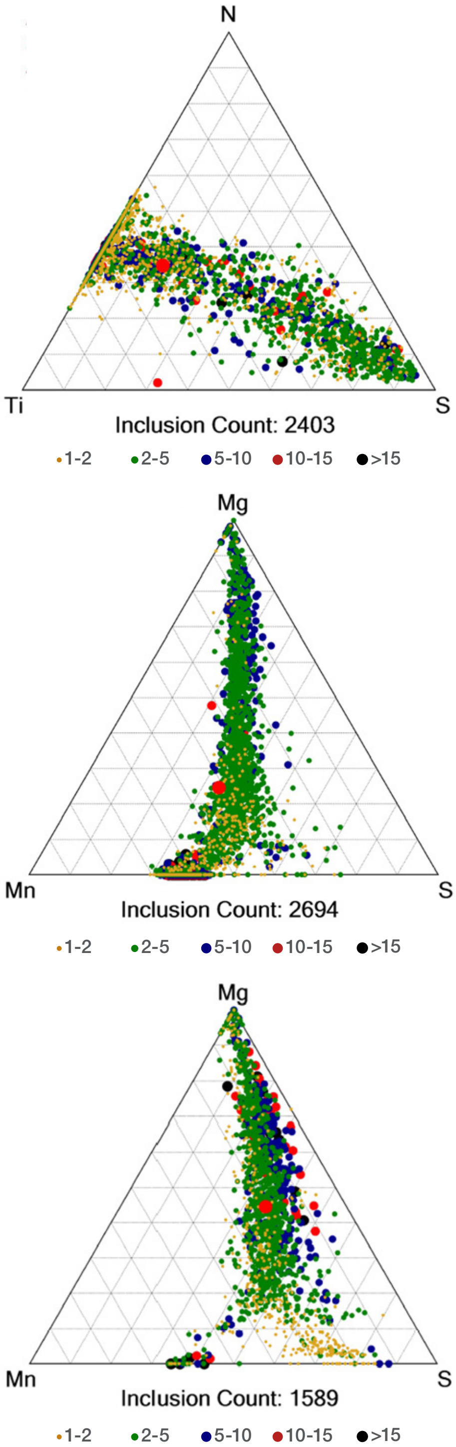

Figure 3. Ternary diagrams of Ti-S-N (top) and Mn-S-Mg (middle) after alloy additions of titanium and magnesium, and Mn-S-Mg (bottom) after calcium addition in the RH degasser.

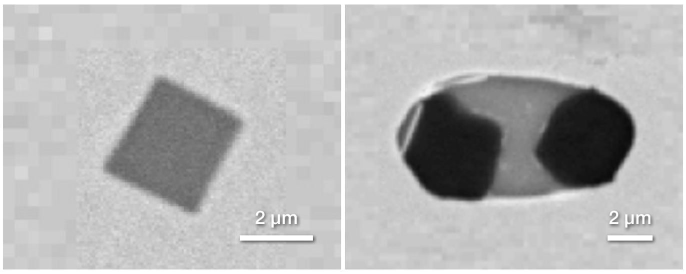

Figure 4. Backscattered electron images can be saved for every inclusion. Here, we show images of a TiN cube (left) and compound MgO-CaS (right), with MgO having a much darker backscattered electron video level. The scale bars are both 2 µm long.

Following the ladle refining was the RH degassing process. Here, the steel was circulated in and out of the ladle through a vacuum chamber situated above the ladle, which readily homogenizes the bath temperature and composition. Here, calcium wire was added after 10 minutes of degassing to modify the sulfide inclusions.

The calcium treatment modifies many of the sulfides by creating CaS, where before there was mostly MnS. The MnS features are identified here at ~40% sulfur on the bottom axis, where the CaS-rich features are closer to the sulfur vertex. There is a continuous distribution of particles between the first phase MgO and the second phase CaS.

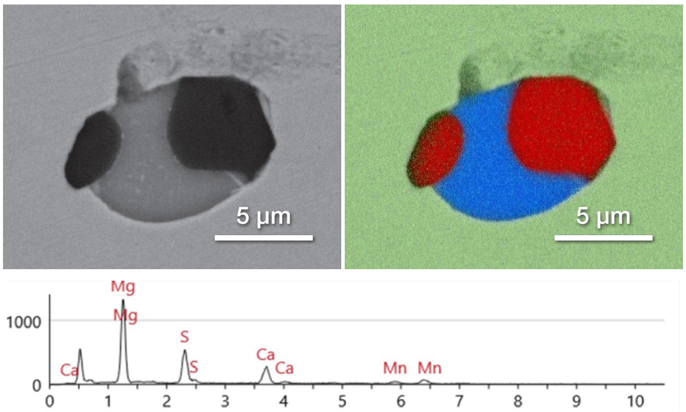

Figure 5. Manual analysis of a compound MgO-CaS inclusion showing the BSE image (left), EDS map (right), and inclusion spectrum (bottom). The map shows Mg in red and Ca in blue with a background of Fe in green.

The Phenom ParticleX Steel Desktop SEM enables fully automated inclusion analysis on the samples. Use of pre-defined recipes and analysis parameters makes the setup swift. With the press of a button, analysis starts and inclusion data is collected—and can be trusted the first time. The collected data can then be easily visualized in a report tailored to your needs.

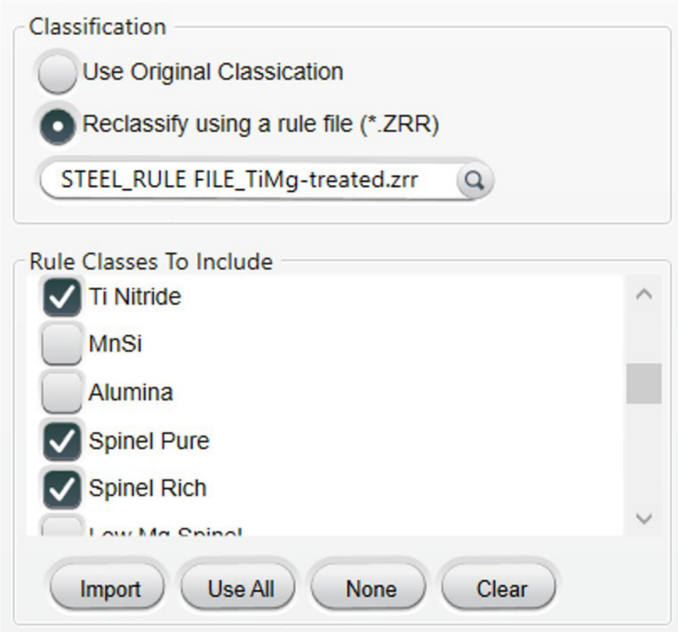

Figure 6. Snapshot of Perception Reporter Software shows that inclusion types may be incorporated in a table, chart, or ternary diagram by selecting the desired classes.

The Perception Reporter software on the Phenom ParticleX Steel Desktop SEM is what makes reporting easy. A unique rule classification can be made by the user, and ternary diagrams can be created with only the desired particle types. The diagrams shown in this report were selected to highlight the inclusion composition changes during ladle refining.

Conclusions

A novel steel deoxidation scheme was studied with the Phenom ParticleX Steel Desktop SEM. Inclusion populations were characterized in polished steel samples from the molten metal refining process. Particle images, compositions, and ternary diagrams were easily reported with Perception Reporter Software. Steel refining with titanium, magnesium, and calcium additions formed TiN, MnS, MgO, and CaS non-metallic inclusions, as well as any combination of the same. As a potential alternative to aluminum deoxidation, this scheme produced non-metallic inclusions that are not prone to agglomeration while also producing TiN, which can be effective at grain refining.

Phenom ParticleX Steel analysis of inclusions

Duration 1:05

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