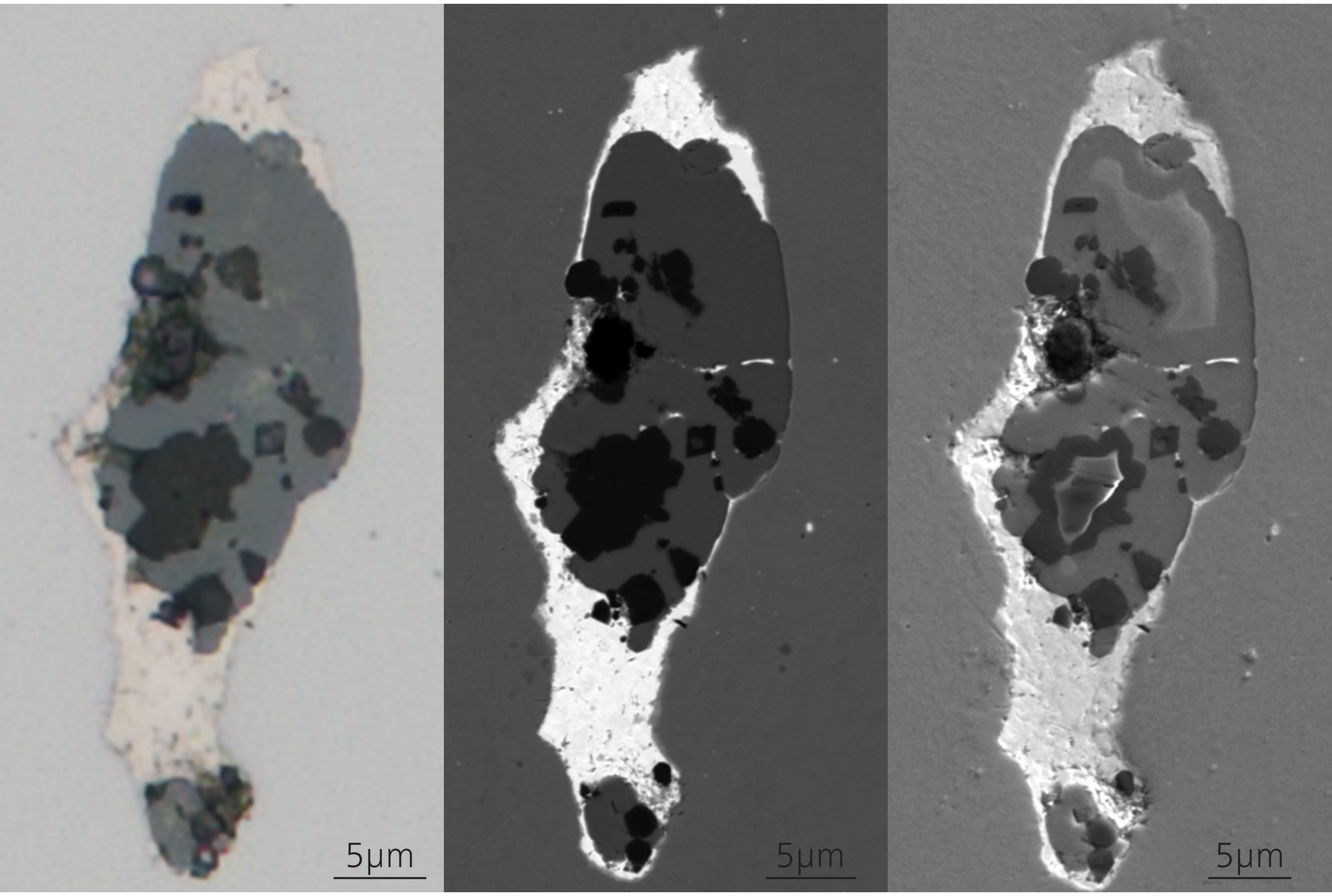
White Paper




# Analysis and Quantification of Non-metallic Inclusions in Steel

Shuttle & Find

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# Analysis and Quantification of Non-metallic Inclusions in Steel

## Shuttle & Find

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**Introduction**

Steel is one of the most important structural materials and its global production has shown a tremendous increase over recent years. In 2007, approximately 1.344 billion tons of steel were sold in the marketplace with new manufacturers entering the market to meet the growing demands. To ensure that the steel fulfills its application requirements, quality control has gained importance as the market offers a wider range of products than in the past.

One of the basic quality issues in steel production is "cleanliness". During the manufacturing process different influencing variables may cause impurities within the product. These predominantly non-metallic inclusions may originate from the covering slag on top of the cast or the lining of the furnace. It is also common that inclusions are generated by chemical reactions with alloying elements, precipitating gases or contaminating elements from recycled scrap metal.
The type and amount of non-metallic inclusions significantly affect the mechanical and physical properties of the steel, e.g. tensile strength, toughness or fatigue limit, and may cause critical failure of the entire part.

In this respect, one of the most important inspection methods is based on the assessment of the microscopic content of non-metallic inclusions in steel using light microscopy (LM). This evaluation is regulated in detail by a number of international standards, notably ASTM E45, DIN 50602 and the new European standard EN10247. However, only the latter is specifically designed for fully automated analysis by LM techniques and allows the rating of inclusions found in modern steel grades, e.g. mixed or colored inclusions.

**Workflow Task**

The common inclusion types comprise sulfides, oxides and silicates that can be identified by their color, shape and appearance in the LM. It is, however, not possible to make a statement about their exact chemical composition. Furthermore, modern steels increasingly contain non-metallic inclusions that cannot be assigned to either of the "typical" inclusion categories. Thus, a vital interest for quality control is to be able to reliably identify any of these conspicuous inclusions. This can be achieved by scanning electron microscopy (SEM) and associated x-ray element analysis techniques.

The challenge is, however, to precisely relocate the small inclusions in the SEM, which were previously identified in the LM, while it is essential to maintain an efficient and user-friendly workflow at the same time. The common practice is to mark conspicuous inclusions manually e.g. by drawing a circle with a marker around the inclusion on the sample surface and thus contaminating the sample surface. This challenge can be overcome by using Correlative Light and Electron Microscopy (CLEM).

Carl Zeiss developed a CLEM solution based on a special specimen holder with three fiducial markers defining a coordinate system that can be calibrated very quickly and semi-automatically with the AxioVision Shuttle & Find software module. The software module itself integrates seamlessly into the established AxioVision user interface and is able to communicate directly with the SEM control unit.

**Instrumentation and Results**

The actual task was to manually inspect the microscopic content of non-metallic inclusions in a mechanically polished steel sample (16MnCr5 grade) at 100 x magnification in the LM. The sample was first mounted into the CLEM "Specimen Holder CorrMic Mat Universal A", which in turn was fitted on the motorized scanning stage of the light microscope Axio Imager.Z2m. The coordinate system of the sample holder was then calibrated using the AxioVision Shuttle & Find module.

As expected, the majority of the non-metallic inclusions could be classified as elongated sulfide stringers and mostly unaligned oxides (Fig. 1). During the inspection process, however, several conspicuous inclusions were detected that appear to be rather large mixed inclusions surrounded by an unidentified third phase. Digital color images of the conspicuous inclusions were acquired with ZEISS AxioCam HRc at 500x magnification (Fig. 2). Regions of interest (ROIs) were subsequently defined in the LM images with the Shuttle & Find software module for further investi-gation with SEM techniques.

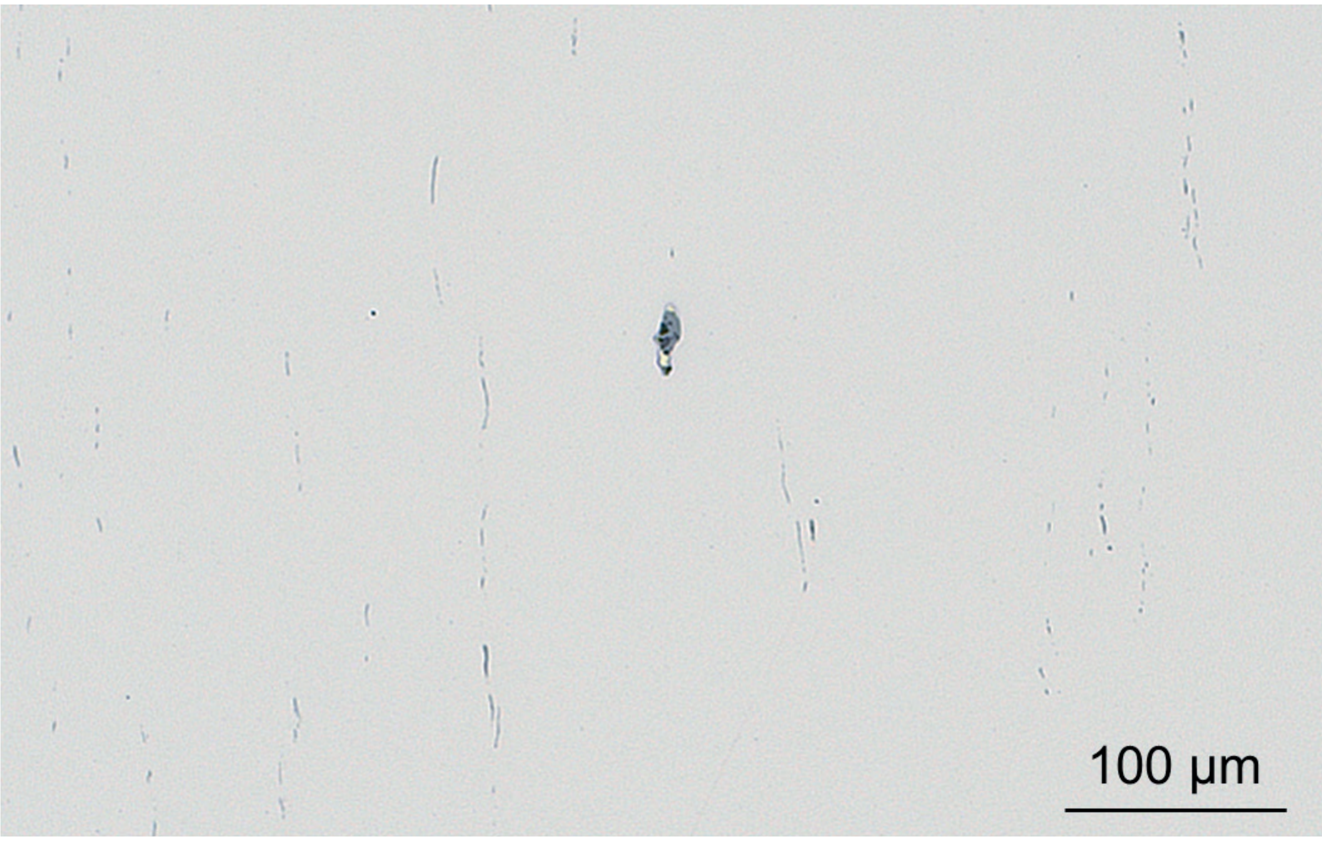


**Figure 1**

*LM image of 16MnCr5 steel grade showing elongated and aligned sulfide inclusions (grey) and small globular oxide inclusions (black). Note that the rather large conspicuous inclusion in the center of the image consists of at least three different phases.*


*Figure 2*

*LM image of 16MnCr5 steel grade showing a conspicuous inclusion stringer.*

*Note that the particles consist of at least three different phases (clear white and different shades of grey).*

The specimen holder was then transferred to a ZEISS FE-SEM for detailed morphological analysis of the inclusions at high magnification. After re-calibrating the holder coordinate system and simply loading the LM digital image with its associated ROIs into the Shuttle & Find module, the selected areas were relocated quickly and precisely in the SEM where the sample was imaged using the back-scattered (BSE) and secondary electron (SE) detection (Fig. 3).

The chemical composition was analyzed by EDS mapping (Fig. 4 and 5). For each inclusion two mappings are displayed to visualize different elements contained in the particular phases.

The resulting element distribution maps confirm that the core of the investigated mixed inclusions indeed contains the primary inclusion constituents MnS and $Al_2O_3$. It was, however, surprising to notice that the unidentified phase is mostly made up of Bi, minor amounts of Cu and P. According to the chemical composition data sheet of this steel grade, only P is allowed in minor quantities, whereas Bi and Cu should not be present. It could thus be proven that the analyzed steel sample is out of specification. As to the origin of these elements, it is most likely that they represent contaminating remnants from recycled scrap metal that was added to the primary melt.

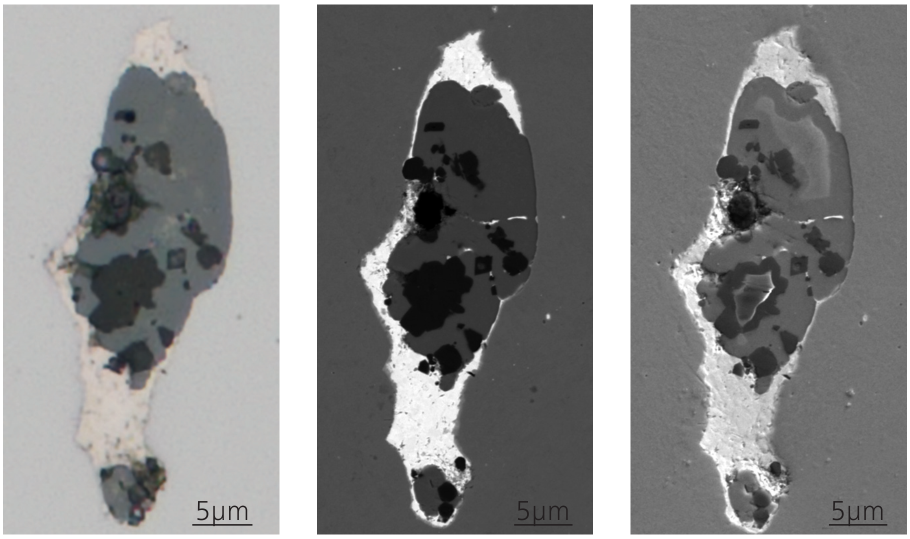


**Figure 3**
*Brightfield image in LM (left) as well as BSE (center) and SE (right) images in SEM of the conspicuous mixed inclusion shown in Fig. 1. Images were acquired using the CLEM technique with the Shuttle & Find module. Note that the bright phase surrounding the core has a rather high density as can be deduced from the BSE image.*

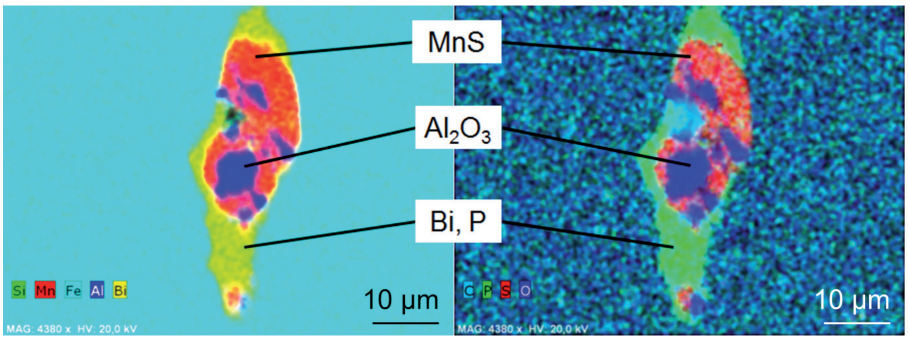


**Figure 4**
*EDS mapping of the conspicuous mixed inclusion shown in Fig. 3. The core of the mixed inclusion consists of the "typical" inclusion types MnS (red) and $Al_2O_3$ (blue) whereas the surrounding bright phase contains the elements Bi (yellow) and P (green).*

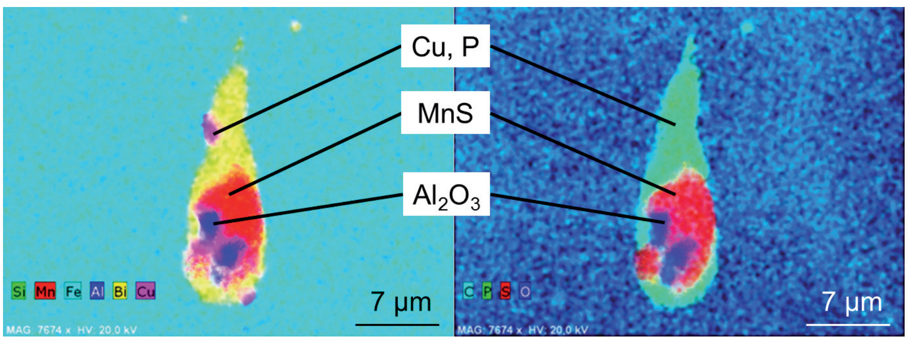


**Figure 5**
*EDS mapping of the conspicuous mixed inclusion shown in Fig. 2 (left most particle). The core of the inclusion consists of the "typical" inclusion types MnS (red) and $Al_2O_3$ (blue) whereas the surrounding bright phase contains the elements Bi (yellow), Cu (pink) and P (green).*

**Conclusion**

Shuttle & Find bridges the worlds of efficient LM and high resolution SEM for the Carl Zeiss product portfolio and adds a significant additional value to the analysis of non-metallic inclusions in daily routine tasks.

As such, automated LM analysis, e.g. with Axio Imager.Z2m and the AxioVision NMI module, allows for a fast and efficient scanning of large sample areas in true color as required by the standards. The rating of non-metallic inclusion content according to five international standards is possible in only one single analysis run and thus a cost and resource efficent screening of polished steel samples can be achieved. Conspicuous inclusions found during the inspection process can easily be documented in the LM and further morphological analysis can be collected in the SEM by applying the CLEM technique. This enables high detail structural imaging and precise information about the inclusion's chemical composition and crystallographic orientation by X-ray analysis techniques in the SEM like EDS, WDS, or EBSD. Therefore, this efficient and streamlined workflow significantly increases productivity and sample throughput.

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