Application Note

Detection, Quantification and Advanced Characterization of Non-metallic Inclusions in Steels

ZEISS ZEN core Non-metallic Inclusions

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Introduction

Non-metallic inclusions (NMI) are present in all grades of steels and related alloys to a greater or lesser extent and can be divided into two categories. Indigenous inclusions occur within the metal itself, as a product of chemical reactions within the melt. These reactions may occur with alloying elements, gaseous species or with impurities introduced from recycled material. Exogenous inclusions are caused by the entrapment of external non-metallic material; this may include pieces of refractory linings or dross and slag materials. Non-metallic inclusions may be oxides, sulfides, silicates, nitrides or a number of other non-metallic compounds, furthermore each inclusion may contain multiple different phases.

Despite non-metallic inclusions comprising a small fraction of the total steel volume, they have a major effect on the mechanical properties. Inclusions can have positive effects, (e.g. improving machinability in free-machining steels) but far more often have negative effects. Detrimental effects may include reduction of dynamic fracture toughness, lowered impact energy absorption and reduced local corrosion resistance, making determining the inclusion type, content and distribution critical to ensure good performance in service.

Advances in steel technologies have drastically reduced the inclusion content of most steels below levels required by corresponding technical standards/specifications, but there remains a need for efficient routine inspection to detect, quantify and characterize inclusions and verify cleanliness of the steel produced according to international standards. The most common inclusion types are oxides, sulfides and nitrides. These most critical inclusions are differentiable by their gray level, color and shape by light microscopy alone, and their exact chemical composition and crystallography can be determined in more detail by electron microscopy. Non-metallic inclusions that do not fall into the above categories are not necessarily covered by existing standards, and while they can often be located by light microscopy the researcher may combine light microscopy with scanning electron microscopy (SEM) with energy-dispersive X-ray spectroscopy (EDS) mapping. This allows full characterization of all inclusions in the steel, for developing improved products and processes

Non-metallic inclusion analysis by light microscopy

ZEN core software environment for multi-user industrial laboratories

ZEISS ZEN core (”ZEN”) is a modular software environment enabling full control of ZEISS light microscopes with advanced image analysis and reporting functionality, combined with user management capability designed for routine microscopy tasks in multi-user industrial quality systems.

ZEISS ZEN core Non-metallic Inclusions is one module available as part of a comprehensive suite of metallographic analysis modules, forming a complete package for metallurgical characterization of steels, including grain size, multiphase, layer thickness, graphite and dimensional measurements.

The overarching concept of ZEN is the adaptive user interface, which enables a supervisor to generate a tailored environment for the operator. Unrestricted, a user can operate in Free Mode for the greatest degree of flexibility where all analytical and microscope control tools are available.

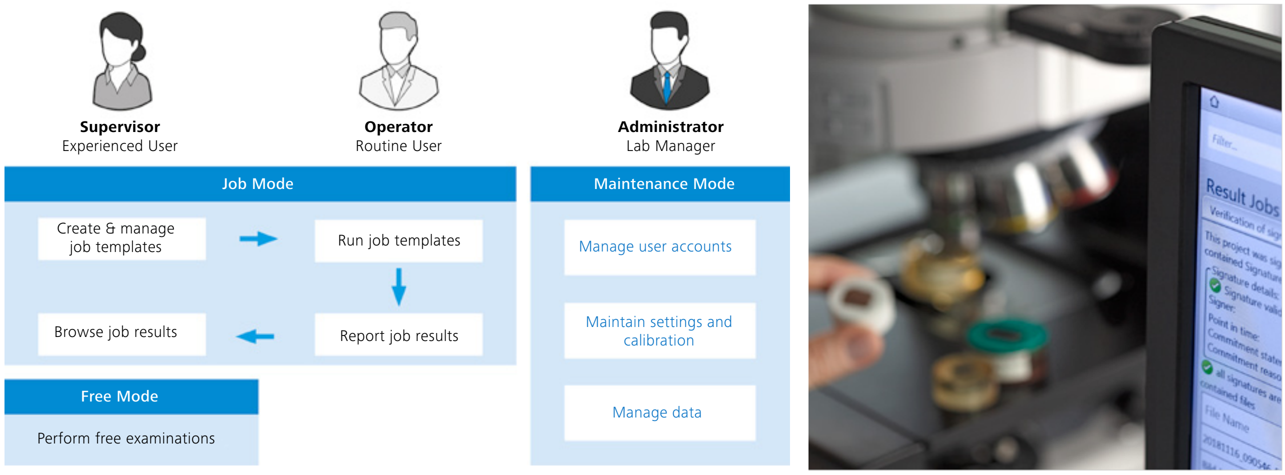


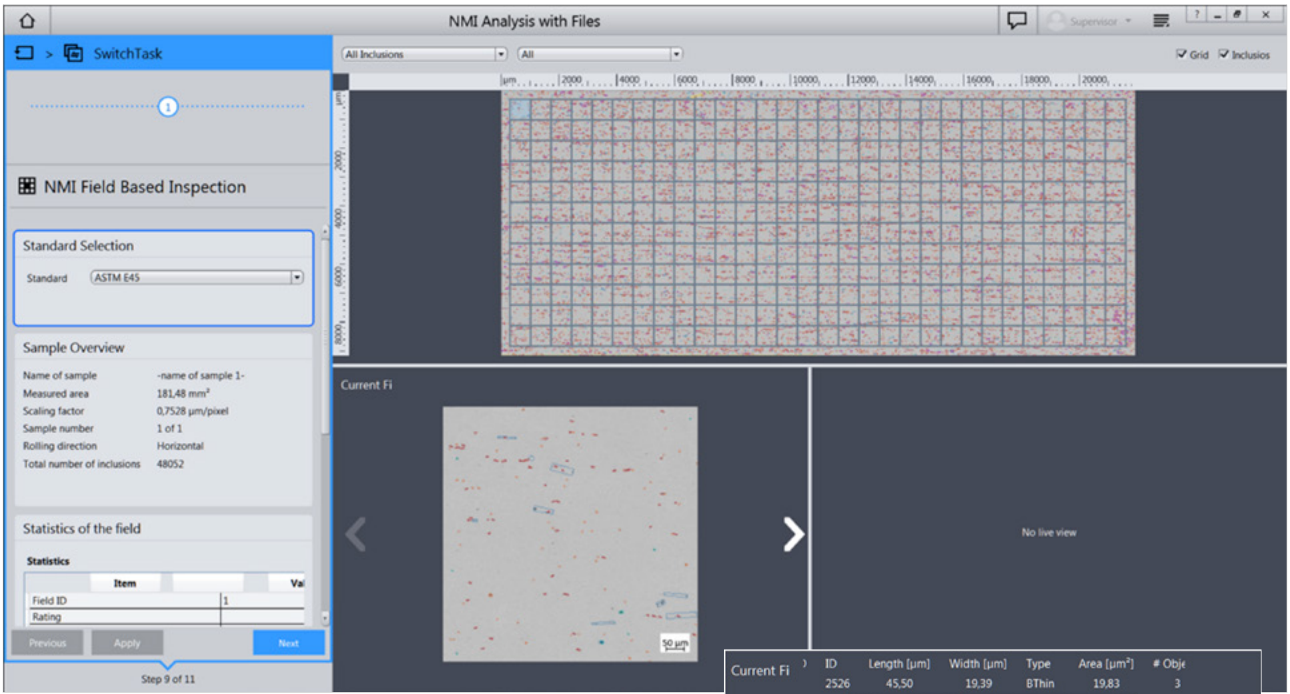
Figure 1 *Adaptive user interface uses three modes to tightly control workflows and permissions according to user groups. In Job Mode, the operator can only change settings necessary for the job. With the optional ZEISS ZEN GxP traceability module, all images, results, analyses and changes are logged and digitally signed by the operator while automatically creating an audit trail.*

Supervisors can create their own workflows and configurations for bespoke analysis, or tailor existing ones to requirements. However, the supervisor may determine that operators may use only Job Mode, with pre-defined straightforward workflows for non-metallic inclusion analysis. These would enforce complete control of hardware and software settings for ultimate consistency. As a result, user-independent reproducible results can be generated in an industrial quality control environment, at any time of day or night, without supervision. The adaptive interface will present only the options authorized, and other settings may “run silent”, meaning they are locked and the operator does not see them and cannot change them.

With the optional ZEISS ZEN GxP traceability module, all images, results, analyses and changes are logged and digitally signed by the operator while automatically creating an audit trail.

Powerful and automated routine non-metallic inclusion analysis

A polished cross-section of a steel sample was examined using an Axio Imager.Z2m compound light microscope equipped with a ZEISS EC Epiplan-Neofluar 10x/0.2 objective. A large surface area (typically up to 200mm^2) was automatically imaged as a series of fields. These are stitched together using the proprietary ZEN stitching algorithm to ensure that inclusions covering multiple fields are measured. By segmenting the image using grayscale values, oxide and sulfide inclusions are automatically identified. An additional module is included, which uses color segmentation to identify nitride inclusions. A gallery of all relevant inclusions is generated, and the user can examine any or all inclusions individually (updating inclusion categorization if warranted) plus view the most severe inclusions in a given field or given sample. Every step of the straightforward workflow is guided, with clear easy-to-follow instructions given at each step.



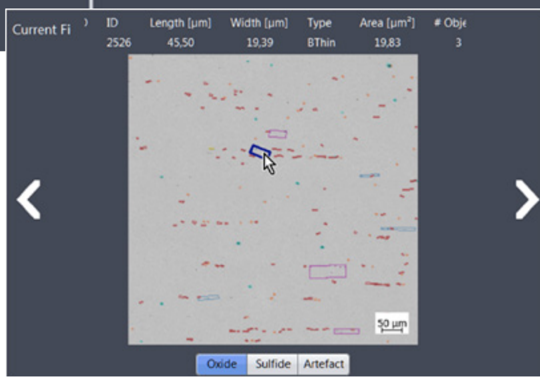
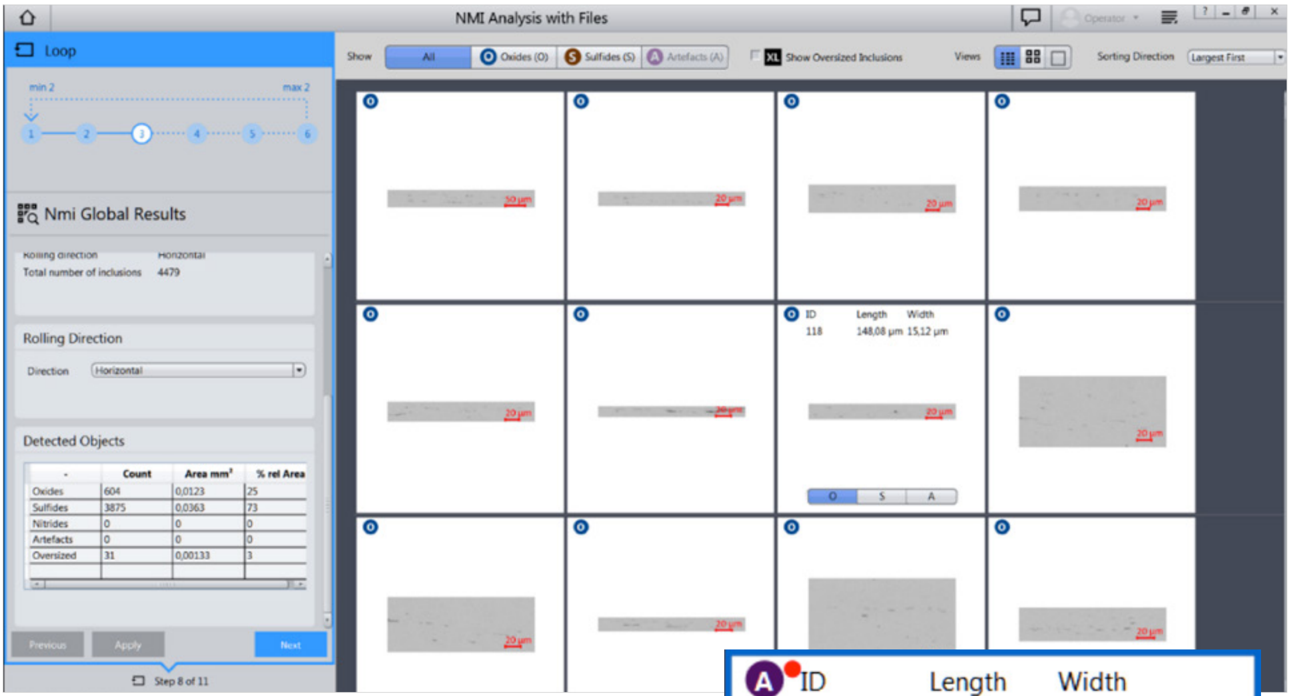


Figure 2 *Field-based inspection of a stitched image of a large area of steel allows rapid oversight of inclusion content and enables live automated navigation to specific fields and inclusions.*

The ZEN core NMI module offers fully automated threshold setting for image segmentation and therefore detection and classification of different species of inclusion. Users are able to benefit from the new Automatic Rolling Direction Detection feature, making setup easy and assisting in rapid understanding and interpretation of results, making NMI analysis fast, reliable and intuitive.

The unique field-based inspection capability (Figure 2) takes the entire stitched image, filters by inclusion type and highlights each class using a different color, and allows intuitive selection and inspection of fields of interest. This feature also allows the user to survey and reinspect certain inclusions in real time. At the click of a mouse, the software automatically drives the microscope stage to the precise field or selected inclusion for deeper free examination of inclusion type and morphology. Statistics can be presented by whole region or a selected field, as well as reported according to the standards.



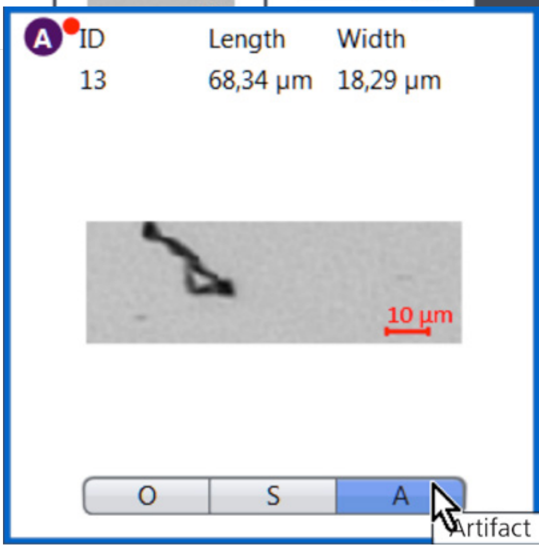


Figure 3 *Inclusion gallery of all detected inclusions, allowing the user to filter by type, obtain detailed statistics on each inclusion or class of inclusions, and if necessary, remove artifacts from statistics.*

Rapid inspection of inclusions is facilitated by the inclusion gallery (Figure 3), able to filter by inclusion type, provide statistics of the product fingerprint, and allow easy closer inspection of individual features via a mouse rollover. In exceptional circumstances inclusion identification can be revised (if required and if user permissions allows) to reclassify detected features. These circumstances may include sample preparation artifacts, contamination on the sample, or after further inspection of a mixed oxide/sulfide inclusion.

The steel sample was characterized according to the ASTM E45 Standard Test Method for Determining the Inclusion Content of Steel[1]. A detailed report is automatically generated (Figure 4) for every sample showing the inclusions detected, their sizes and all necessary analysis and sample information. Inclusion analysis can be carried out according to a broad range of standards, including ASTM E45, DIN 50602, EN 10247, GB/T 10561, ISO 4967, JIS G0555 and SEP 1571. There is no need for multiple separate analysis runs – a single run can give a report for each separate standard without the need to re-image. Reports are archived in a searchable format, containing all necessary images of significant inclusions and can be exported freely.

Evaluation Report

Detailed result per specimen

ASTM E45 / Method A (worst field)

Characteristic values / Inclusion type	A Thin	A Heavy	B Thin	B Heavy	C Thin	C Heavy	D Thin	D Heavy	Ds Thin	Ds Heavy	Dos Thin	Dos Heavy
Total inclusion count	154	0	11	4	38	0	4	0	2	0	0	0
Total inclusion length [µm]	1,436.5	0.0	265.6	116.6	585.5	0.0	13.9	0.0	6.6	0.0	0.0	0.0
Severity Level	3.5	0.0	1.5	1.0	2.5	0.0	1.0	0.0	0.5	0.0	0.0	0.0
ASTM E45 / Method A												
Specimen ID	1											
Measured area [mm²]	24.70											

Inclusion Id	**688**
Global type	**Sulfide**
Specific type	**α**
Length [µm]	**33.85**
Width [µm]	**4.91**

Inclusion Id	**1664**
Global type	**Nitride**
Specific type	**EFB**
Length [µm]	**191.02**
Width [µm]	**56.1**

Inclusion Id	**1284**
Global type	**Oxide**
Specific type	**EB**
Length [µm]	**106.4**
Width [µm]	**24.44**

Figure 4 *Extracts from example report on NMI inclusion analysis generated by ZEISS ZEN core NMI.*

User management concept enables ultimate traceability and data integrity

Quality assurance and certification are becoming increasingly important in metals manufacturing with demands for both 'built-in' quality and robust audit trails becoming stronger, particularly for structured standards such as those for non-metallic inclusion measurement. Internal and external audit and certification require continuous improvement in quality systems, and metallography workflows are a key component of product specification and certification. Traditionally, microscopic analyses have not readily been traceable and controllable; however, the user management concept in ZEN core opens new possibilities in control and recording of metallography workflows.

Originally developed for the highly regulated pharmaceutical industry, the ZEISS ZEN core module GxP is an optional module that enables a steel manufacturer or industrial core facility to adhere to the greatest levels of user control, data integrity and auditable reporting within routine microscopy workflows.[2] The module ensures that supervisors can set well-controlled workflows for non-metallic inclusion measurement that conform to international standards. Operators can only access and change settings relevant to their permitted jobs. Every single relevant interaction between operator, microscope and software is recorded all while providing a detailed encrypted audit trail and preventing data tampering or falsification. This gives the user full control of routine analysis, protection from manipulation of results and a clearly traceable record of actions taken in collecting a result.

Advanced Correlative Inspection of Inclusions

Much information about inclusions can be gained from light microscopy, including inclusion size, shape, distribution and general categorisation. However, to characterize an inclusion as completely as possible, it is necessary to use electron microscopy techniques as seamlessly as possible.

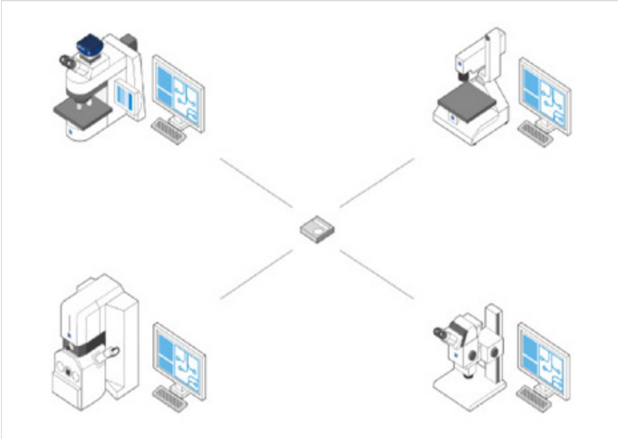


Figure 5 *Correlation and data connectivity between multiple different microscopes using Shuttle & Find and ZEN Connect.*

This is facilitiated by Shuttle & Find, ZEISS's system for automatic correlation of features between different microscopes and scales. The sample is loaded onto a correlative light microscopy holder, and any features of interest located on the light microscope can instantly be re-located when the sample is transferred to the electron microscope. Aligning with this, ZEISS has developed the ZEN Connect data connectivity solution which offers the ability to overlay data from multiple sources and scales to correlate microstrutural imaging, morphological, chemical and other data at multiple resolutions for rapid interpretation of multiple data streams. Furthermore, the ZEISS ZEN Connect system allows the user to visualise all sample data from all microscopes in a single intuitive and simple map interface, from a wide view zooming down to the tiniest details – keeping data from multiple sources together. This is illustrated in Figure 5.

In this way, a particular inclusion can be identified in the light microscope, rapidly located under the electron microscope, and analysed further by the researcher to assess its morphological, chemical, crystallographic and other characteristics.

Following examination of an advanced high strength steel (FB590) by light microscopy, a number of small inclusions were observed, generally <5µm in size (Figure 6). These appeared to have an inhomogeneous composition. High resolution brightfield light micrographs were sufficient to automatically locate these inclusions and determine (based on shape and color) that they were largely oxide based. The specimen holder containing the sample was then transferred to a ZEISS Crossbeam 540 SEM for more detailed morphological and compositional analysis of the inclusions at much higher magnification. Using recalibration of the holder coordinate system and loading the LM image with associated regions of interest into the Shuttle & Find module, regions of interest can be quickly and precisely relocated in any ZEISS scanning electron microscope and can then be imaged and characterized using the wide array of available detectors and spectrometers.

The chemical composition was analyzed by EDS mapping with the most pertinent elements detected shown in Figure 6. The inclusion center was clearly oxide based, but both aluminum and magnesium were detected, indicating that this was likely a mixed Al_2O_3/MgO inclusion or a magnesium-aluminum spinel. The oxide core was surrounded by a calcium sulfide shell, and two titanium rich regions were observed on either side of the inclusion, which may indicate small titanium nitrides/carbonitrides or possible contamination from the preparation process. This indigenous inclusion is probably the result of calcium treatment of the steel, during which sulfur in the steel melt is bound to oxide/aluminate particles as CaS, and thus not deposited at grain boundaries as sulfide-based inclusions.

The unique connectivity of ZEISS systems means that this can all be represented as a single data set using ZEN Connect. Further examination of the same regions is possible by advanced techniques such as focused ion beam scanning electron microscopy and X-ray microscopy for 3-dimensional mapping of the inclusion, its composition and the surrounding region.

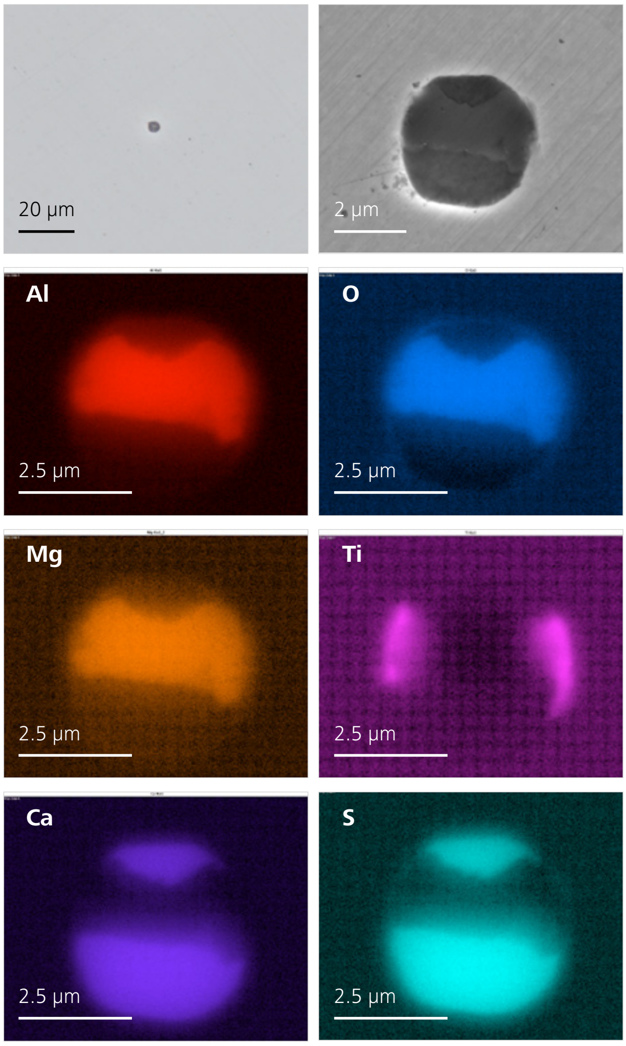


Figure 6 *Very small inclusion (<3µm) in FB590 dual phase steel, located using brightfield light microscopy then, using a correlative workflow, examined in a ZEISS Crossbeam 540 at 15kV using secondary electron imaging and EDS mapping.*
Images courtesy of James Russell, AIM Facility, University of Swansea.

Conclusions

Automated light microscopy analysis using the ZEISS ZEN NMI module in ZEN core allows a fast and efficient study of the inclusions present in any steel, following all relevant major inclusion analysis standards. Significant inclusions found during inspection can be documented and investigated rapidly and consistently in a multi-user environment thanks to the adaptive user interface and advanced user management tools available within the ZEN core software. After location and identification in light microscopy, specific inclusions can be further analyzed by the researcher or quality analyst in scanning electron microscopy with EDS. This gives structural mapping and compositional information at high resolution, critical in interpreting results for product and process improvement. The user control and recording principles in the software makes possible unprecedented levels of reproducibility for metallography as part of a steel producer's total quality management system.

Shuttle & Find and ZEN Connect enhance this further by bridging light microscopy and efficient high resolution SEM/EDS and making it straightforward to move samples between the two, automatically relocating regions of interest. Combining NMI analysis with the full portfolio of material analysis modules such as grain size analysis, the GxP module for auditability and traceability, and the power of multimodal connected microscopy, ZEISS ZEN core is a complete metallography solution for the steel manufacturer or core research facility.

References:

[1] ASTM E45-18a, Standard Test Methods for Determining the Inclusion Content of Steel, 2018

[2] Application of GxP principles to routine metallography, ZEISS Microscopy, 2018

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