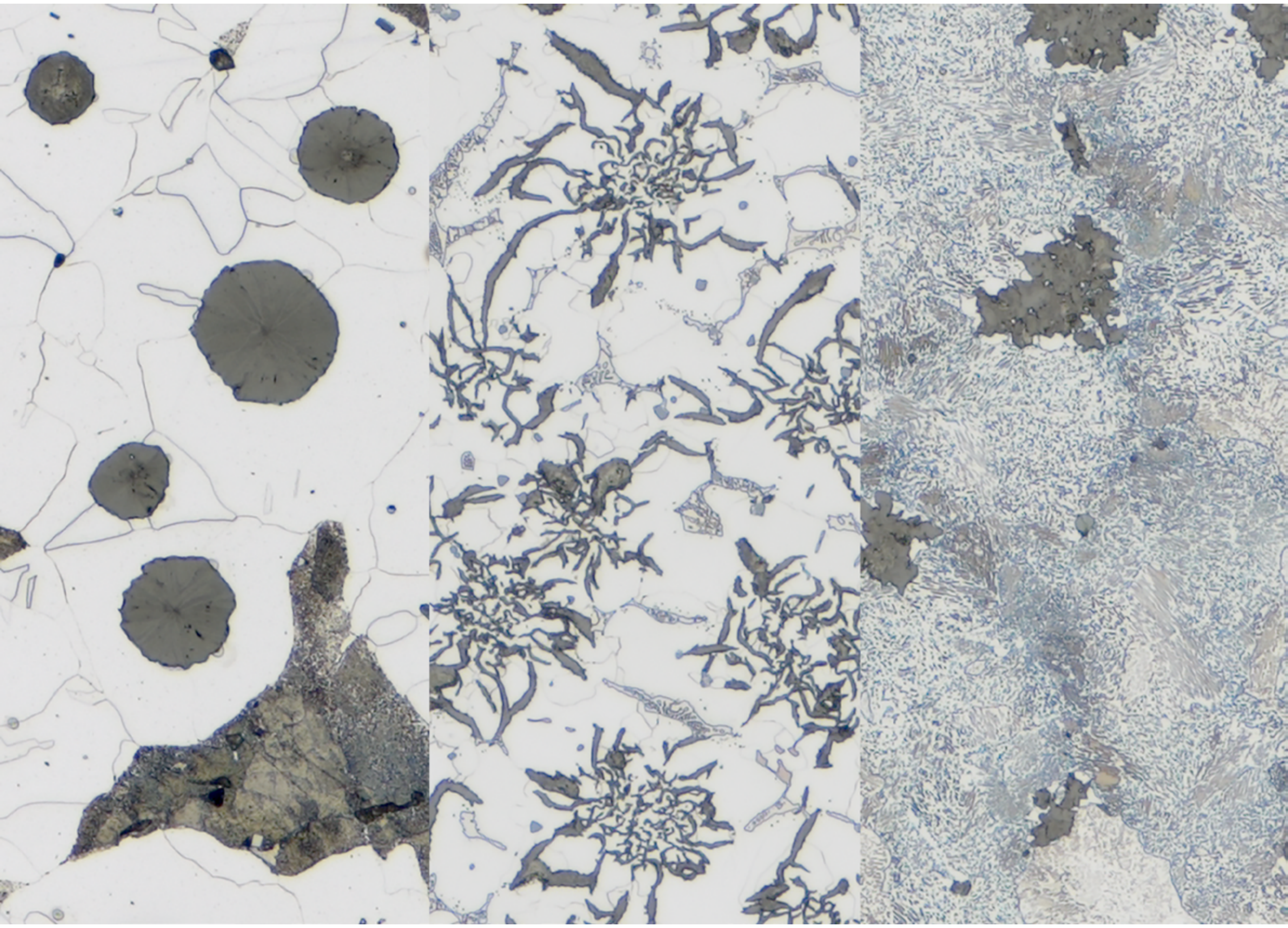
White Paper



# Microscopic Methods in Metallography

Using ZEISS Axio Observer and ZEISS Axio Imager

# Microscopic Methods in Metallography
## Using ZEISS Axio Observer and ZEISS Axio Imager

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**Introduction**

The Aalen Materials Research Institute (IMFAA) at Aalen University studies the application and further development of methods in materialography, in the field of functional composites, magnets, and battery materials. In its research efforts, the IMFAA works with material synthesis and material analysis of traditional metallic and ceramic materials, as well as modern functional materials used in energy technology and composites. Its tasks involve changing samples and changing requirements, the multiple factors influencing the research questions posed, and the search for a microscopic solution that also meets requirements for flexibility.

Knowledge of the structure, crystal structure, and micromorphology, as well as elemental composition and distribution are essential for ensuring safety in designs and components of all sizes – ranging from fields of mechanical engineering and aeronautical engineering to power station engineering and electrical engineering. The observed structures, which are responsible for the properties of the materials to a significant extent, are becoming increasingly smaller and therefore more difficult to differentiate. Microscopic testing is therefore both a necessity and a challenge at the same time (Fig. 1). In many cases, a combination of light microscope and electron microscope is required to clearly differentiate the structures. However, the metallographic microscope is still the instrument of first choice for analysis. The microstructure is visualized by conventional light microscopy following metallographic preparation. The samples are sectioned, wet-ground and polished with a diamond or oxide suspension until they are free from scratches or deformation, then (optionally) chemically etched to make particular microstructural features visible. The structures are being made visible by conventional preparation with etching and impression methods.

For most materials, final chemical or electrolytic etching is necessary to make the structure visible. Some materials are also suitable for illumination, e.g., using polarized light as a contrast method (“optical etching”).

***Figure 1*** *Microstructural analysis of macrosections of larger nonferrous cast components. The inverted design of the microscope makes it easier and quicker for users to conduct their analysis. Samples do not need to be sectioned and mounted; those being inspected are positioned directly at focus level, allowing even large and heavy samples to be examined directly.*

Examples of typical metallographic applications are presented in the following sections.

**Grain Size Determination – ASTM E112 on Steel for Electric Strip**

Grain size and distribution have a significant effect on the material properties and are sensitive parameters in the metallurgy of these materials. It is especially easy and quick to determine the grain size using comparative overlays; see Figure 2. However, appropriate software (ZEISS ZEN core) can also be used to measure the grain size on polished and etched microsections quickly and reproducibly.

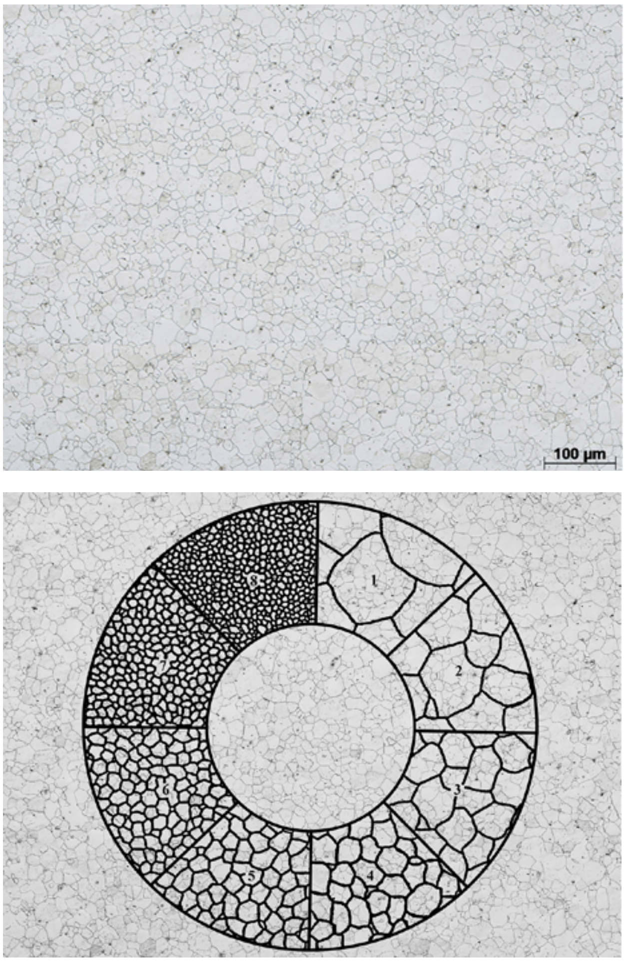



***Figure 2*** *Non-alloy, low-carbon steel of high purity. Above: Steel microstructure, showing ferrite grains. Below: grain size comparative overlay as per ASTM E112. Etching: 1% HNO3. 100× magnification. (Objective: EC Epiplan-NEOFLUAR 10×/0.25 DIC; brightfield)*

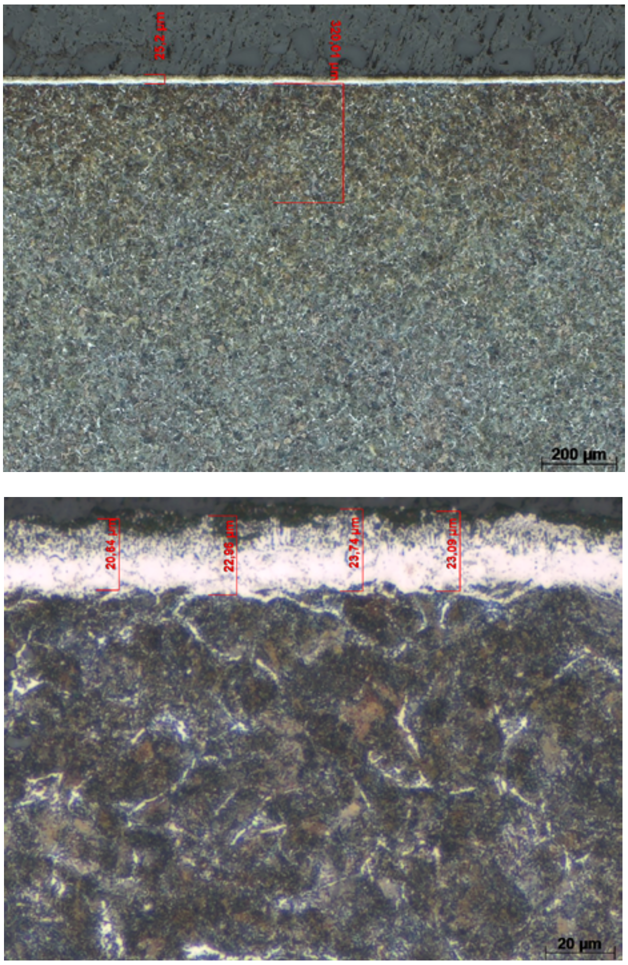



***Figure 3*** *TENIFER® nitrided case; compound layer (25 µm) and diffusion layer (320 µm). Matrix: heat-treated structure made of tempered martensite with individual nitride deposits. Etching: 3% HNO3. Above, 50× and below, 500× magnification. (Above: objective: EC Epiplan-NEOFLUAR 5×/0.14; below: objective: EC Epiplan-NEOFLUAR 50×/0.55 HD DIC, brightfield)*

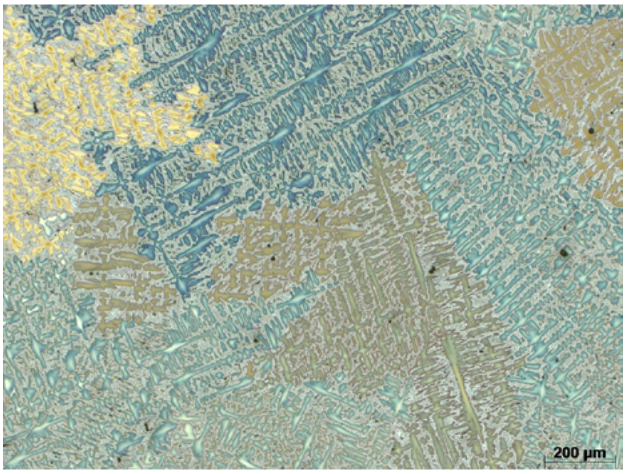
### Layer Thickness Determination on a TENIFER® Nitrided Case

Nitrocarburization following the TENIFER® method is used to increase the surface hardness, resistance to wear, fatigue strength, and resistance to corrosion of the materials. These are usually nitriding steels that are especially suitable for this approach. Layer thickness and nitriding hardness depth are significantly influenced by the composition of the materials; both parameters are a measured variable for assessing the technical production process. Figure 3 shows a typical nitrided case. The thickness of the compound and diffusion layer is measured at varying magnifications.

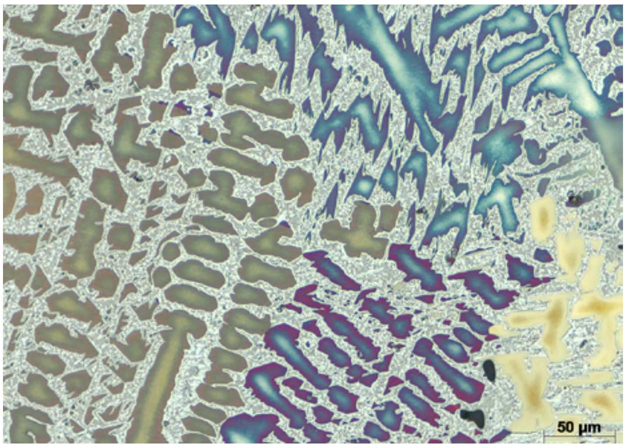
### Evaluation of the Structure of Nonferrous Metals in Bell Bronze

Revealing the solidification structure to determine homogeneity across the component or measure the grain size allows conclusions to be drawn about the quality of the bell metal cast allow conclusions about the quality of the bell metal cast. For example the sound properties are influenced by the alloy, the structure and the porosity. In the case of bearing bronzes with a lower tin content, the quantity of eutectoids is important for the wear properties of bearing alloys. Figure 4 shows the structure after color etching. Depending on the magnification, certain characteristics can be differentiated and measured.

**Cast grain size 50×**




**Mixed crystal and eutectoid 200×**




**Eutectoid 500×**

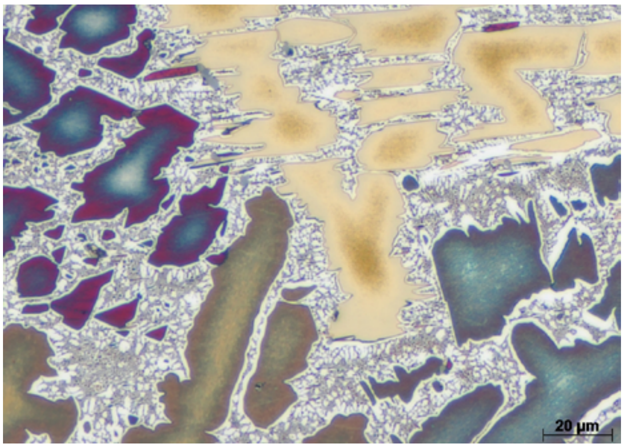



***Figure 4*** *Bell bronze; dendritic α-mixed crystals in different crystal orientations; α+δ-eutectoid in remaining fields.*
*Etching: Klemm III. 50×/200×/500× magnification.*
*(Objectives: EC Epiplan-NEOFLUAR 5×/0.13 DIC, EC Epiplan-NEOFLUAR 20×/0.50 HD DIC, EC Epiplan-NEOFLUAR 50×/0.80 HD DIC, brightfield)*

### Test and Quantification of Structure Development in Special Brass Alloys

The process of revealing the structure of special brass alloys, in conjunction with analysis, is mainly used for quality control purposes. The effect of a rolling texture of the α-mixed crystal phase with an overlaid texture of silicide deposits is important in the processing stage. The size, quantity, and distribution of the silicide precipitations primarily affect the sliding and wear properties of the material. Figure 5 shows such a heterogeneous structure.

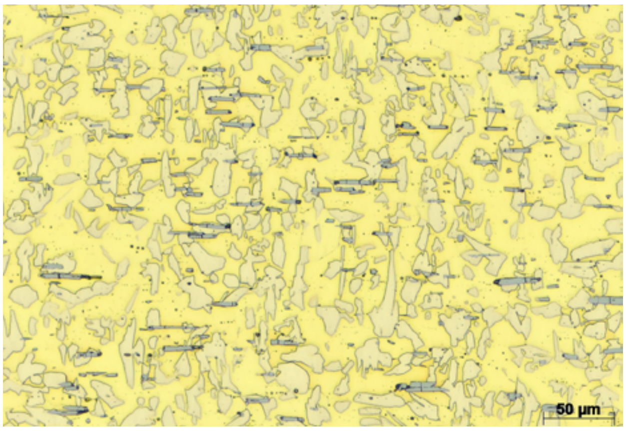



***Figure 5*** *Special brass: matrix of ß-mixed crystal with oriented α-crystals. Silicide precipitation structure oriented in rolling direction.*
*Etched and polished with 10% ferric nitrate. 200× magnification.*
*(Objective: EC Epiplan-NEOFLUAR 20×/0.50 HD DIC, brightfield)*

### Quality Assurance of Structural Steel Beams

By revealing the normalization structure and examining the formation of the banded structure conclusions can be drawn about properties. Checking the formation of the banded structure allows conclusions to be drawn about properties such as workability, weldability, tendency toward lamellar fracture, and other such characteristics. To produce load bearing constructions, protection against brittle fracture and absence of cracks is essential for the welding process of steel. Directly on the component, several points are electrolytically polished and etched without a great deal of metallographic preparation. They can then be directly analysed with ZEISS Axio Observer. Figure 6 shows the structure of this type of steel beam.

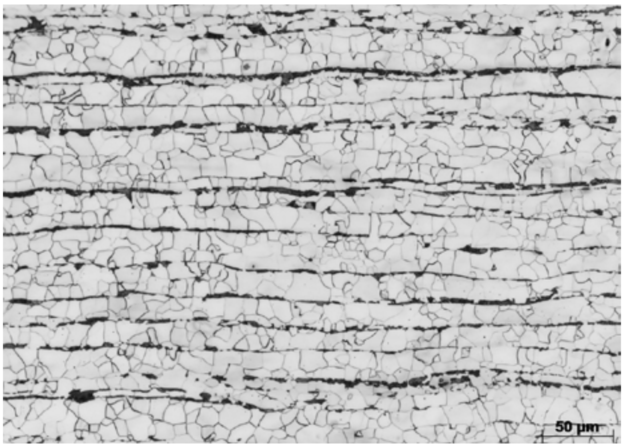



***Figure 6*** *Longitudinal section of structural steel. Formed banded structure made of ferrite (bright) and perlite (dark) due to normalized rolling. Etching: electrolytically etched with A2. 200× magnification. (Objectives: EC Epiplan-NEOFLUAR 20×/0.50 HD DIC, brightfield)*

**Quality Control of Aluminum Casting**

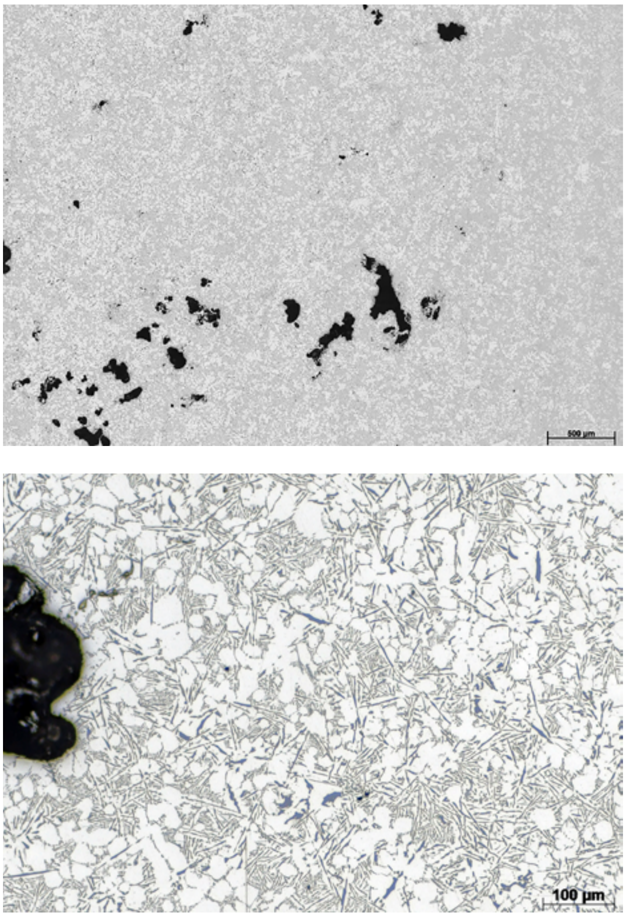



***Figure 7*** *Aluminum silicon cast alloy; hypoeutectic alloy with microshrinkage and micropores. Points of varying fine and coarse formations of eutectic. Unetched. Consolidated image. Top: 100× magnification taken with Tile Image; Bottom: 100× magnification. (Objective: EC Epiplan-NEOFLUAR 20×/0.50 HD DIC, brightfield)*

Pores in cast aluminium are clearly made visible, and their quantity, size and distribution can be measured. Varying cooling speeds in different regions of the component can be seen using a large-surface high resolution scan of the sample.

**Porosity Measurement of 3D-Printed Metal**

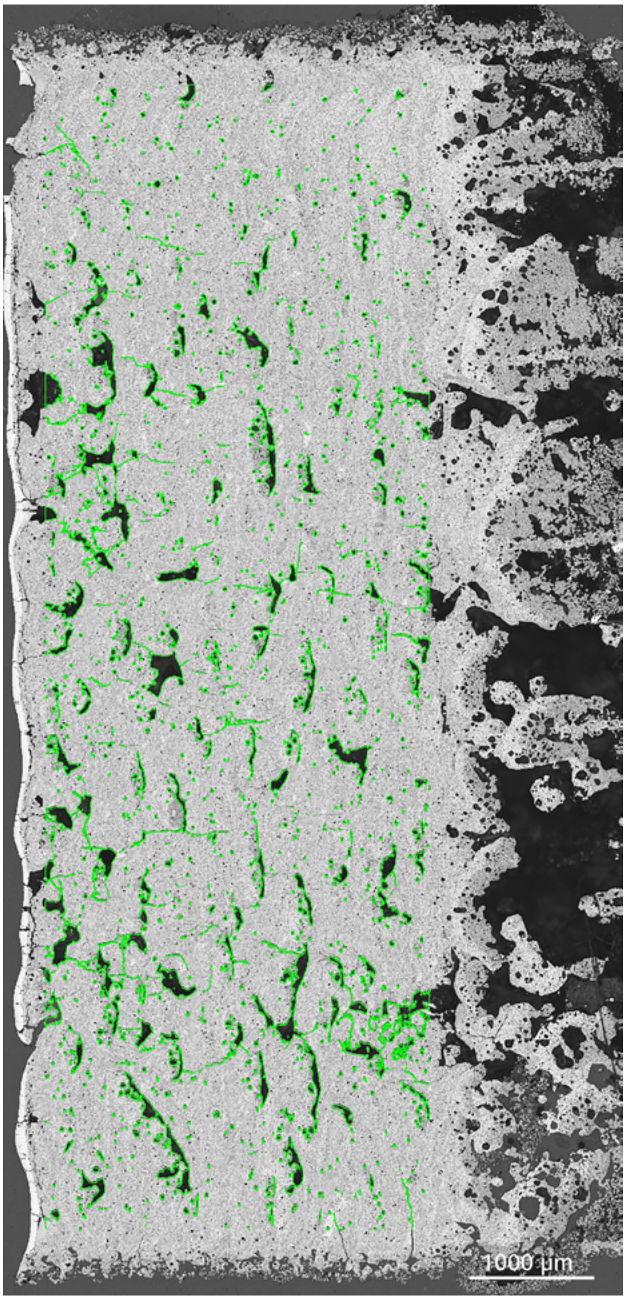



***Figure 8*** *Laser-fused component made of metal. Production-dependent porosity allows conclusions to be made about the production parameters. Unetched. 100× magnification. Consolidated image. (Objectives: EC Epiplan-NEOFLUAR 20×/0.50 HD DIC, brightfield) Tile Image (8×6)*

The porosity of components from 3D-printed metal depends on the composition of the source powder and the laser settings. Quantitative analysis of porosity (Figure 8: Pores detected in green) allows conclusions to be drawn about the production parameters.

**Phase fraction determination of duplex stainless steels using machine learning**

Austenitic stainless steels are tough, relatively easy to weld but susceptible to stress corrosion cracking whereas ferritic stainless steels are resistant to stress corrosion cracking but comparatively brittle and hard to weld. A duplex stainless steel contains both austenite and ferrite in approximately equal proportions, leading to synergistic interaction and new properties. Duplex stainless steels are used where weldability, corrosion resistance and mechanical strength are necessary.

The austenite to ferrite ratio is affected by composition but also by thermal history, particularly in or near a weld. Determining the ratio is key to validating performance and understanding the local properties. With appropriate etching, austenite and ferrite take on distinct appearances. Using a combination of machine learning (with ZEISS ZEN Intellesis) and multiphase analysis, austenite and ferrite are automatically segregated. This gives an accurate average of the relative volume fractions as shown in Figure 9.

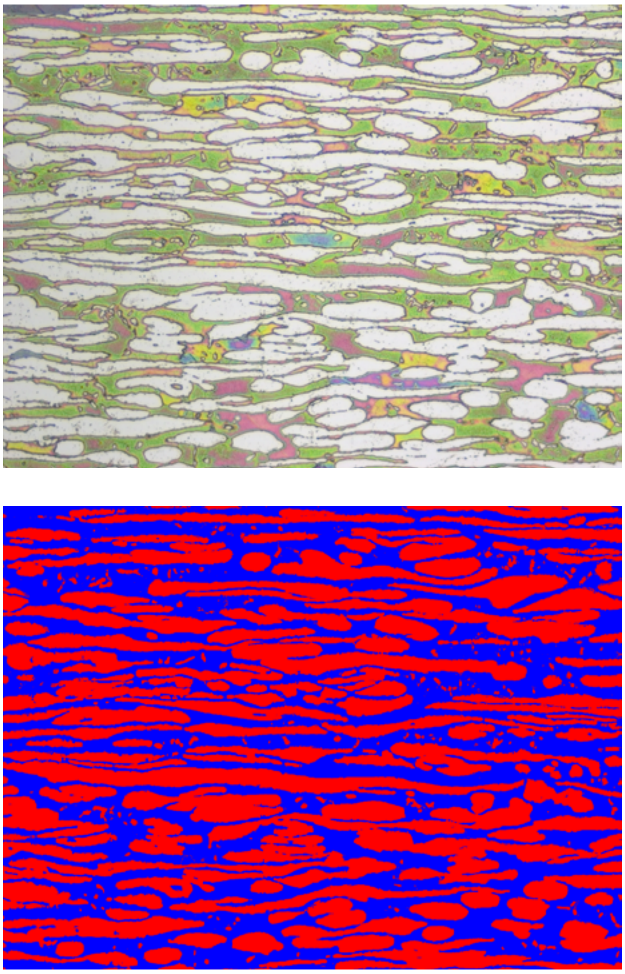


***Figure 9*** *Duplex stainless steel. This micrograph has 47% ferrite (blue) and 53% austenite (red) segregated using machine learning with ZEISS ZEN Intellesis. Sample courtesy of TWI Ltd.*

**Auditability and traceability**

Metallography is the eyesight of the metallurgist. The microstructure, combined with chemical and mechanical testing, forms the basis of acceptance testing of metal products. Metal producers perform a variety of standardized test methods to grade and certify their products. In particular, grain size and non-metallic inclusions are especially critical parameters in assessing the quality and therefore value of finished steel. Customer and independent auditing therefore seeks to verify that the metals quality system is fit and appropriate for the financial exposure and risk to the manufacturer in case of product defect or process failure, particularly in highly regulated industries such as aerospace or medical processes.

Taking grain size as an example – by using the ZEISS ZEN GxP module a user can build in high levels of due diligence into routine microscopy. Individual workflows are defined, conforming to the relevant Standards. All relevant interactions between a human, microscope and software are then recorded, providing an audit trail. This provides full control over routine analysis, protection from manipulation of results and a clearly traceable record of actions taken in collecting a result, as shown in Figure 10.

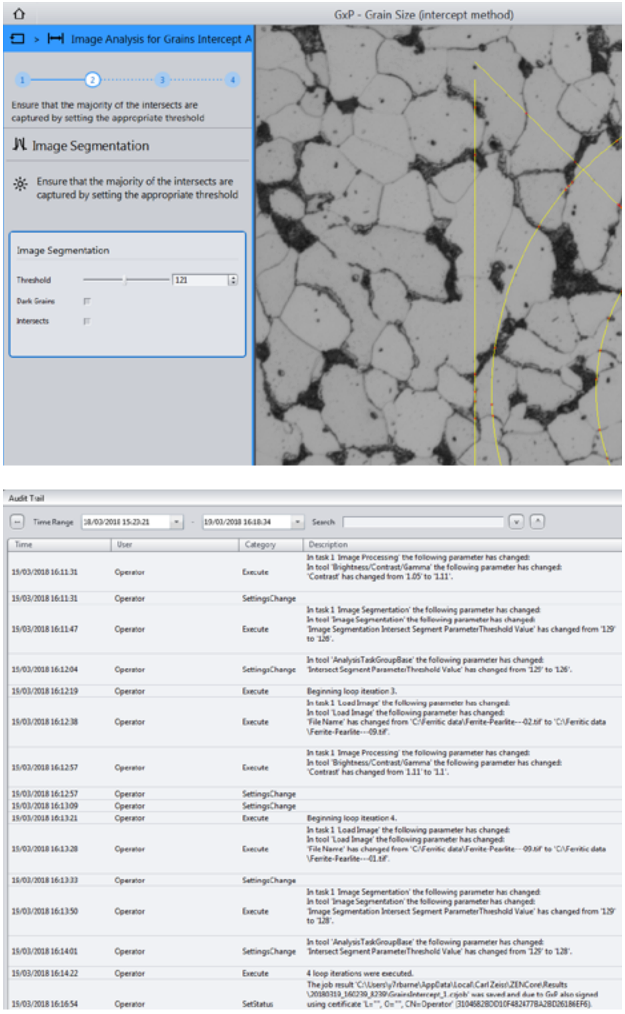



***Figure 10*** *ASTM E112 grain size measurement (using the intercept method) under the GxP module where the operator can only change relevant parameters and a full encrypted audit trail is generated of all inputs and changes.*

**Summary**

For clear and reliable structure interpretation, it is especially important to conduct the microscopic analysis under conditions following the highest requirements. In addition to determining and describing the structure, examination of the materials in question involves, for example, layer thickness measurement, quantitative analysis of phase parts, grain size analysis, or the determination of the degree of purity under existing standards and guidelines.

All of these requirements can be covered perfectly with ZEISS Axio Observer or ZEISS Axio Imager, using the related ZEISS ZEN core analysis software.

The following properties of ZEISS Axio Observer are especially noteworthy:

- rapid movement of sample stage, manually and software-controlled
- easy operation with touch TFT; all functions can be engaged directly
- scattered light through eyepieces can be excluded by switch
- reliable communication from software and microscope
- light manager allows for preset illumination for each objective lens
- Koehler illumination of LED possible using adjustment screws
- Polarization, DIC, or darkfield optionally available

***Figures on cover*** *From left to right: Spheroidal graphite cast iron (GJS), lamellar graphite (gray) cast iron (GJL), and annealed cast iron (white heart malleable cast iron, GJMW). Each one etched with 3% HNO3. 200x magnification. (Objectives: EC Epiplan-NEOFLUAR 20x/0.50 HD DIC, brightfield)*

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