Application Note

Metallic Grain Structures and Their Microscopic Analysis

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Metallic Grain Structures and Their Microscopic Analysis

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Metallic materials are interpreted in terms of their inner structure – known as the grain structure – in addition to a wide range of other chemical and physical properties. This inner structure is referred to as the "macrostructure" or "microstructure" depending on the size of the elements being observed. The grain structure is typically adapted to the technical application of the material and can be viewed with conventional light microscopes, provided that the specimen being examined has been appropriately prepared. This preparation process is also referred to as "metallographic preparation." Depending on the composition of the metal, its processing history (e.g. heat treatments and forming processes) and the expected structural properties (e.g. grain size, content of nonmetallic inclusions), this metallographic preparation involves selecting the mechanical or electrolytic processing procedure as well as the subsequent chemical and microscopic contrasting and enlargement. This text is intended to provide a practical and readily comprehensible insight into this topic.

What is a grain structure and why should it be examined with a microscope?

A metallic grain structure consists of individual, typically microscopic crystalline areas known as "grains." Macroscopic grains are less common and, depending on the base metal, are more frequently found in nonferrous materials (e.g., aluminum, copper, zinc). Grain structures with details that can be seen with the naked eye or a magnifying glass are referred to as macrostructures. If, on the other hand, a microscope (stereo, reflected light or digital) is required to assess the material, the term microstructures is used.

These grains form from the molten material when it solidifies, interact with one another and with "foreign elements" (phases, contamination), and of course also react to subsequent external influences – such as chemical processes (corrosion), chemical and/or physical influences (e.g. heat treatment processes) or purely mechanical influences, such as downstream forming processes. The structure, size and orientation of the grains result from the material composition (alloy) used and the way the material is made (forging, casting, or additive manufacturing).

Once metallographic preparation is complete, visible light can interact with the prepared structural grains and their crystal shapes and boundaries under a light microscope, making them visible. This typically occurs at magnifications of 25x to 1000x, which corresponds to the limits of traditional light microscopy. Lattice defects, structures and elements at the sub-microscopic level (<1 µm) and down to the atomic level are assessed using electron microscopes rather than light microscopes. As such, electron microscopes (SEM, TEM) that can be extended by means of optional element analysis equipment (EDX: e.g., ZEISS EVO 10/15/25; WDX) are used if greater magnifications are required.

Based on the microsopic image, it is then possible to draw conclusions regarding the characteristics of the material or the workpiece (materials that are part of an overall system). For example, the grain size and general microstructure can be used to determine the degree of hardness and resilience; certain phases may cause the material to become brittle or influence its resistance to corrosion. As a result, assessments of a material's microstructure are not only a means of evaluating characteristics for the design and processing stages, but also play a significant role in assessing cases where damage has occurred.

Structural elements that can be evaluated using a light microscope include (Fig. 1):

- Grains/crystallites and their grain boundaries
- Intermetallic phases and precipitates
- Nonmetallic inclusions (NMI) and phases

The evaluation is based on the following criteria:

- Type and form
- Size and number
- Distribution and orientation

Based on all of this information, it is then possible to create a comprehensive description of the microstructure (of the grain structure) and draw conclusions regarding its potential characteristics.

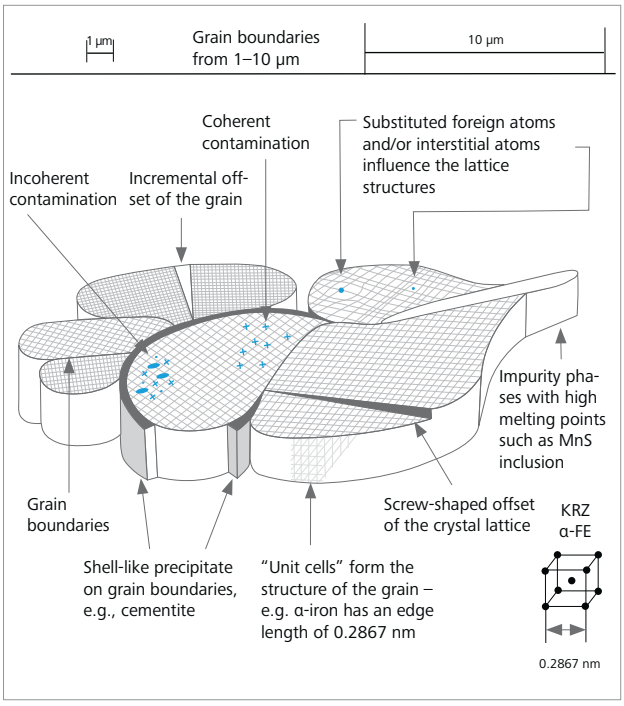

Fig. 1: *Schematic grain structure of a ferrous material*

The materials used in practical applications today are a mixture of various chemical elements sometimes also referred to as "alloys." Steel and cast iron are essentially alloys based on iron (Fe) with carbon (C) alloying additions, which are responsible for the hardness of the ferrous material. The carbon may take the form of a pure element (lamellar and globular graphite in cast iron, Figs. 2 and 3) or may be present as an intermetallic phase, which is also referred to as iron carbide Fe3C or cementite.

In the case of soft, low-carbon ferritic steels, the hard cementite is typically precipitated along the boundaries of the ferritic grain in the form of tertiary cementite or as a small proportion of pearlite (Fig. 4). Iron carbide Fe_3C occurs as a lamella in the harder structural element pearlite and this lamellar structure is made visible following etching. This banding, along with lower reflection of light, make the pearlite appear darker than the ferrite (Figs. 2 and 5).

Fig. 2: *Pearlitic cast iron with lamellar graphite, etched with nital. The carbon is primarily present as graphite in a lamellar form, which results in reduced strength. The pearlitic matrix itself exhibits a sufficiently high degree of hardness.*

Image taken with ZEISS Axio Imager, 50x objective, brightfield

Fig. 3: *Ferritic cast iron with spheroidal graphite, etched with nital. The carbon is primarily present as graphite in a spherical form. The spherical form results in improved strength in comparison to lamellar cast iron, but the hardness of the material is lower due to the lack of cementite in the purely ferritic matrix.*

Image taken with ZEISS Smartzoom 5, at approx. 500x magnification

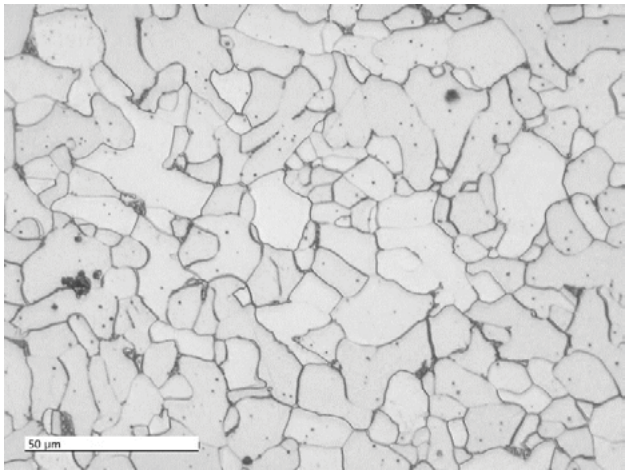

Fig. 4: *Ferritic steel with approx. 0.1 % C, etched with nital. The carbon is primarily present in the form of cementite and as a low proportion of pearlite between the ferritic grains. The matrix, which is therefore nearly purely ferritic, has a low degree of hardness but very good resilience.*

Image taken with ZEISS Smartzoom 5 at approx. 500x magnification, coaxial illumination with low proportion of ring light

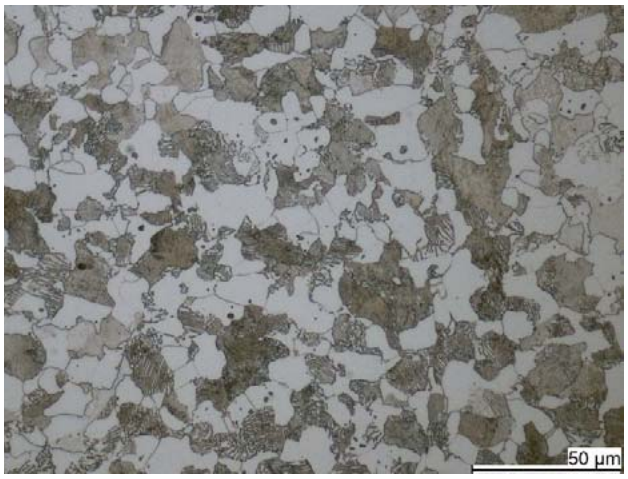

Fig. 5: *Ferritic-pearlitic steel with approx. 0.2 % C, etched with nital. The carbon is primarily present as a cementite lamella in a harder proportion of pearlite adjacent to the ferritic grains. This causes the cementite to appear streaky. The pearlitic grain reflects less light than the ferritic grain and thus appears darker. A matrix of this type has a higher degree of hardness than the steel in image 4, but a lower resilience.*

Image taken with ZEISS Axiolab, 50x objective, brightfield

Other alloying elements may include metals such as aluminum (Al), chromium (Cr), manganese (Mn), vanadium (V), nickel (Ni), molybdenum (Mo) and silicon (Si), which through their inclusion cause the iron lattice to have certain properties. Other nonmetal elements such as nitrogen (N), hydrogen (H), oxygen (O), phosphorus (P) and sulfur (S) are frequently undesirable "steel parasites," as they typically negatively impact the properties of the metal. Their products (e.g. oxide and sulfide) are often analyzed in the course of determining the NMI (determination of non metallic inclusions, also frequently referred to as determination of steel purity). However, within certain limits they can also serve as desirable alloying elements.

How is this matrix made visible?

Metallographic preparation tailored to the material forms the basis for the correct depiction of the grain structure. A wide range of preparation methods can be used depending on the material composition and the question being addressed. There is sufficient literature available regarding metallographic preparations. Furthermore, manufacturers of metallographic preparation equipment and consumables provide procedural documentation on their websites, which can be viewed and downloaded as a guide when getting started.

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Metallography is a type of destructive physical testing. The component undergoing metallographic testing is destroyed in nearly all cases (as in the production of metallographic specimens) or at least slightly damaged. However, it may still be possible to use the component in some cases – such as in the case of on-site component testing. The following section briefly describes the steps involved in producing a specimen for metallographic examination.

The first step is taking the sample. To this end, a wet abrasive cutting process is used to take a representative sample from the workpiece being examined. A thin, rapidly rotating disc containing an abrasive is used for this purpose (Fig. 6). This sample is intended to represent the structural condition of the entire component or a specified area of its structure (welding seam, joints, heat-treated areas). This naturally means that the cutting process should be designed to treat the sample with care, so that it is not subjected to any damage that modifies its structure. This is achieved through the structural design of the cutting discs and equipment, which are tailored to the material and application scenario in question.

The cutting process establishes the surface that will be subsequently examined under the microscope.

Next, an embedding process is used to fix the cut pieces so they can be handled more easily and to standardize their dimensions (Fig. 7). Epoxy or acrylic resin is typically used to fix the pieces; cold (up to approx. 100 °C, at atmospheric pressure or in a vacuum) or warm (at max. 350 bar and 180 °C) embedding press machines can be selected for this step.

Fig. 6: *Wet abrasive cutting machine with clamped gear wheel for taking a sample of a section of a gear tooth. This section, which is typically induction- or case-hardened, will be examined with respect to its structure and hardness and thus needs to be separated from the component.*

Fig. 7: *Variety of embedded samples of varying shapes. Performing embedding with various synthetic resins ensures a good preparation process outcome and makes this procedure efficient.*

While the prerequisites for an ideal preparation and therefore a good representation of the structure are established during the cutting and embedding steps, the grinding and polishing process is likely the most important step in terms of the microscope examination. During this process, the macroscopic roughness of the cut surface is reduced to the point of attaining a reflective surface. If the intention is only to make the macrostructure visible, a few coarser grinding steps and contrasting with acidic or alkaline solutions are sufficient. A reflective surface is not required for this, as the specimen will typically be examined using a stereo microscope (Fig. 8).

Nonetheless, the mirror finish is required to make the microscopic elements of the structure visible under a reflected-light microscope. It is achieved by gently milling the surface with fine to very fine abrasives following surface grinding. Milling continues until nearly all damaged areas are removed from the surface, typically using diamond, aluminum oxide or colloidal silicon dioxide on appropriate polishing cloths and discs. The success of the preparation can typically be checked early on using the differential interference contrast (DIC), which shows even the slightest deformations in the surface (Fig. 9). If DIC is not available, observing the specimen under brightfield conditions is sufficient in most cases. The sample should only be etched after this step. The etching process enhances contrasts that are invisible or only visible to a limited extent under brightfield conditions and allows the microstructure to be examined with a high degree of accuracy. Typically, only weak acids are used for this step. Weak nitric acids with an alcohol content of 1–3 % (nital) are very often used for unalloyed and low-alloyed carbon steel (cf. Figs. 2–5). Corrosion-resistant steel requires special etching processes, such as color etching, which influences the interference of the microscope's light on the surface, or electrolytic etching (Figs. 10 and 11).

Fig. 8: *Welding seam ground on two levels of SiC film, followed by macro etching with 5 % aqueous nitric acid.*

Image taken with ZEISS Stemi 508 stereo microscope at 15x magnification

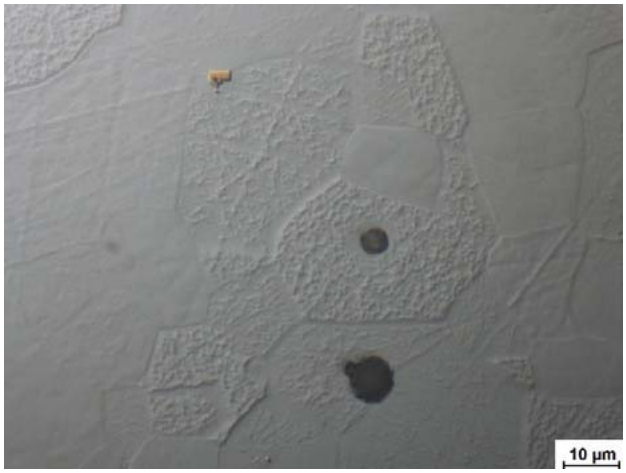

Fig. 9: *Ferritic steel with titanium carbide and oxide inclusions following mechanical preparation to 1 µm diamond. Fine traces of deformations can still be observed in the differential interference contrast image. The sample has not been etched.*

Image taken with ZEISS Axio Imager, DIC, 100x objective

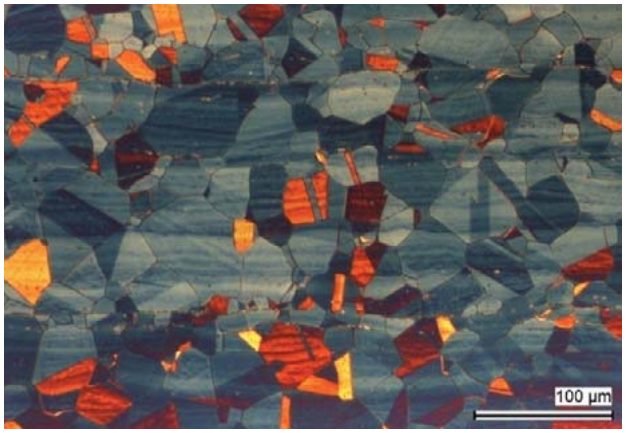

Fig. 10: *Corrosion-resistant austenitic steel after final polishing with OP-S and subsequent Lichtenegger and Bloech color etching. Austenite grains with twins and ghost lines in the direction of deformation become visible.*

Image taken with ZEISS Axio Imager, brightfield, 20x objective

Fig. 11: *Corrosion-resistant austenitic-ferritic steel (duplex) following electrolytic etching in 20 % sodium hydroxide solution. The austenite grains (light brown) are embedded in the bluish-brown ferritic matrix.*

Image taken with ZEISS Axiolab, DIC, 20x objective

Please note: The desired structures only become visible when there is an ideally coordinated interplay between the preparation technique, the appropriate chemical or electrochemical etching process and the microscopic contrasting technique (brightfield, DIC, polarization) as well as the desired resolution (of the smallest discernible feature).

Which contrasting techniques can we now use to characterize the structural properties of metals?

The option of using different contrasts on a light microscope enhances the analysis of metallic structures. This type of contrasting, which can take place either on its own or in conjunction with preceding chemical or electrolytic contrasting procedures, is also referred to as “optical etching.” The following section discusses the most common contrasting techniques used for routine analysis of structures and provides examples of several typical applications.

Technique	Application	Measuring	Documentation
Reflected light brightfield *(Fig. 12)*	Standard (e.g., grain boundaries, nonmetallic inclusions, phases, fissures, oxidation)	●	●
Reflected light darkfield *(Fig. 13)*	Lacquer and plastic coatings, composite materials, glasses, corrosion products	●	●
Reflected light DIC *(Fig. 15)*	Deformation, protrusions		●
Reflected light polarization contrast *(Figs. 16 and 17)*	Grain size, grain shape and grain orientation following electrolytic etching (Barker) for Al and Al alloys; grain structure in unetched condition with hexagonal lattice types (e.g., Co, Ti, Mg, Zn, Zr)	●	●
Reflected light fluorescence *(Figs. 18 and 19)*	Fissures, pores (when open during embedding)	●	●

Table 1: *Examples of applying contrasting techniques for examining metallic structures*

The **brightfield** is a standard technique for all types of material analyses. Fissures and pores, nonmetal phases and oxidation products are first observed in an unetched condition, as they typically exhibit different reflective behavior than the base metal material. The location of fissures and pores in relation to other structural characteristics, on the other hand, can typically only be evaluated if appropriate chemical etching has been carried out (Fig. 12).

The **darkfield technique** is used less frequently in metallurgical microscopy than it is in the microscopy of nonmetal materials. However, this contrasting technique also offers several advantages for metals, as well as when evaluating colored structures such as layers of lacquer or plastic coatings on metal substrates. This contrast can also be used to evaluate corrosion products (Fig. 13). Darkfield microscopy can be used to show very fine scratches on polished samples as a method of examining the grinding quality.

Fig. 12: *Laser welding seam on high-alloy steels with fissures and pores following electrolytic etching. These are also visible in the unetched condition, but the intercrystalline course of the fissures can only be evaluated after etching has been completed.*

Image taken with ZEISS Axio Imager, brightfield, 5x objective

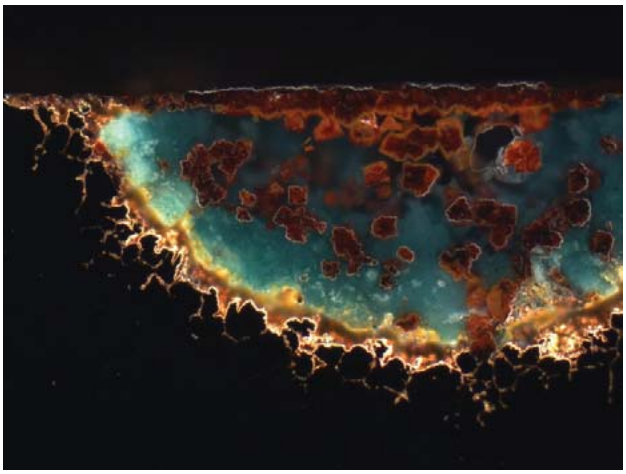

Fig. 13: *Corroded area on a brass pipe, unetched. Reflective areas appear dark (metal substrate) under the darkfield microscope, while the corrosion products can be observed in their own color.*

Image taken with ZEISS Axio Imager, darkfield, 20x objective

The **differential interference contrast** is a useful tool for analyzing very fine deformations that may still be present in the surface after polishing (cf. Fig. 9). Checking the quality of polishing prior to the etching process can prevent additional effort being required during preparation. DIC can also be used to distinguish hard and soft structural elements, as hard phases are removed to a lesser extent than softer ones during the final polishing process. They therefore "protrude" from the surface (Figs. 14 and 15).

This minimal difference is not typically visible under a brightfield microscope, but can been seen in DIC. As such, this contrast can be used to make a qualitative distinction between the hardness of different phases. A further key advantage is the possibility of making grain structures such as grain boundaries visible even in an unetched condition (Fig. 15). The structure can therefore be evaluated prior to etching, so chemicals that pose a risk to health do not need to be used on materials that are difficult to etch (corrosion-resistant materials). However, a perfect final polish is required in this case.

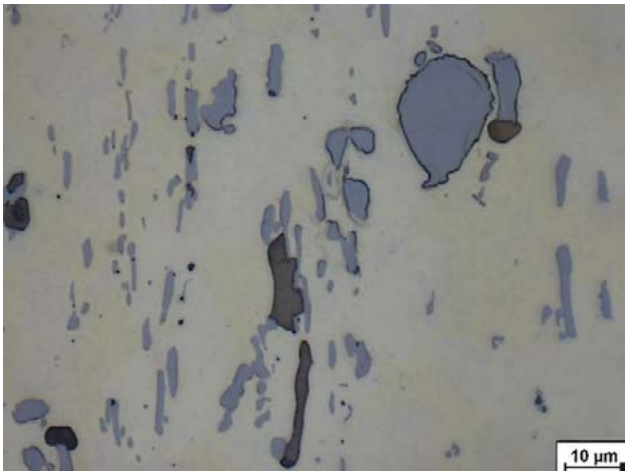

Fig. 14: *Copper alloy after final polishing. Due to their reflectivity, the various phases appear to have different colors under a brightfield microscope.*

Image taken with ZEISS Axiolab, brightfield, 100x objective

Fig. 15: *Copper alloy like that in Fig. 14. Due to their ablation behavior, the phases of varying hardnesses have varying heights, which are only visible in DIC microscopy. This enables a qualitative distinction between their hardnesses. At the same time, the grain structure can already be made visible in the unetched condition.*

Image taken with ZEISS Axiolab, DIC, 100x objective

The **polarization contrast** is primarily used in the analysis of materials with a hexagonal lattice structure. This includes titanium, zinc and magnesium in particular, as well as a range of other materials (Fig. 16). However, aluminum and its alloys can also be analyzed under polarized light after being etched appropriately.
This requires electrolytic etching with tetrafluoroboric acid, which is known as Barker etching (Fig 17).

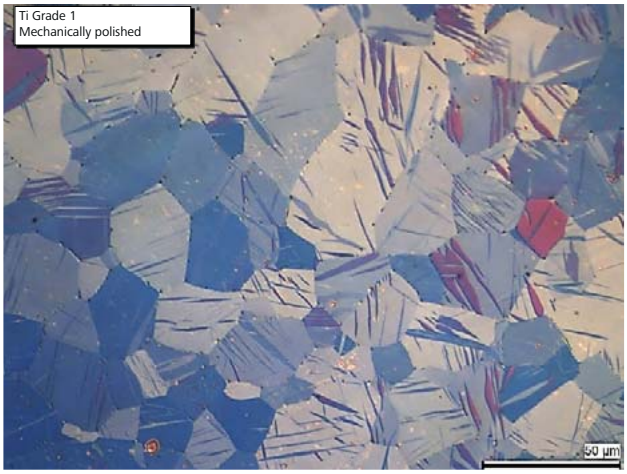

Fig. 16: *Technically pure titanium (Grade 1) following mechanical polishing, seen under a polarization contrast microscope. Unetched surface. The polarized light is enhanced or eliminated on the crystal faces due to the hexagonal lattice structure, which manifests itself as a contrast between light and dark. The image appears in color due to a so-called λ/4 plate.*

Image taken with ZEISS Axio Imager, polarization contrast, 20x objective

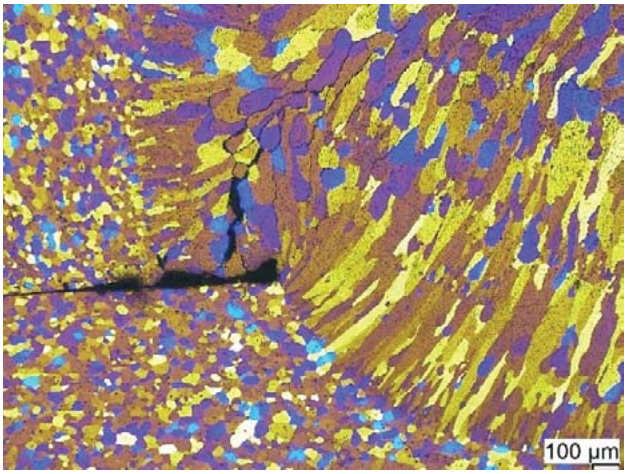

Fig. 17*: Aluminum welding seam following electrolytic etching with tetrafluoroboric acid (Barker etching), seen under a polarization contrast microscope. The etching creates a layer of oxide of varying thickness depending on the orientation of the crystals; the polarized light can interfere in this oxide layer. This also results in elimination and enhancement effects.*

Image taken with ZEISS Axio Imager, polarization contrast, 5x objective

Fluorescence is a further contrasting technique that can be used in metal and material microscopy. This method exploits the fact that certain materials that are excited at a certain wavelength emit visible light at another wavelength. These fluorescent powders (e.g., Epodye) are mixed with the embedding agent (typically transparent epoxy resin) during the embedding process and, together with it, penetrate existing and open pores and fissures. This procedure is supported by vacuum impregnation. Following curing and preparation, the microscope's light in the blue spectrum excites the fluorescent dye, which then emits light in the yellow-green spectrum. The filled pores or fissures are illuminated in yellow-green (Figs. 18 and 19).

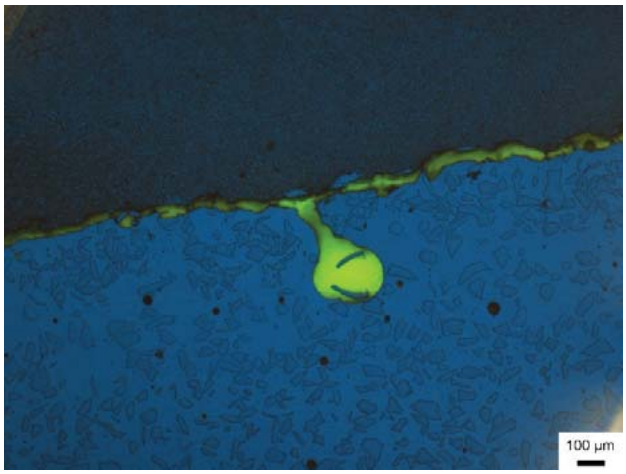

Fig. 18: *Pore and fissure between a WSC coating and the steel to which it is applied. This is illuminated in yellow-green in the corresponding microscope contrast because the fissure was penetrated by embedding agent with fluorescent powder. The fissure was therefore present prior to embedding and may have arisen during fabrication; however, it could theoretically also have occurred during the cutting process and be a preparation artifact.*

Image taken with ZEISS Axio Imager, fluorescent contrast, 5x objective

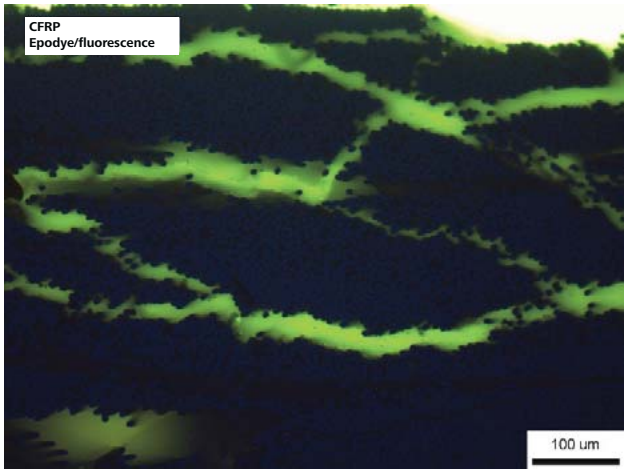

Fig. 19: *Fissures in a carbon fiber composite material*

Image taken with ZEISS Axio Imager, fluorescent contrast, 20x objective

The light microscope equipment

Reflected-light microscopes are used to visualize structures in the range from mm to approx. 1 µm (10x/20x/50x/100x objectives are typical for the material microscope. Fig. 20). The design can be either upright or inverted, which is advantageous for large specimens or when viewing clamped specimens in specimen holders. High-definition stereo microscopes, on the other hand, are used less frequently for structural interpretations. The image generated by the microscope must be rendered faithfully and be suitable for integration into current documentation systems.

Because the illumination and contrasting of the reflective samples is performed by the imaging optics – the microscope objective – special requirements apply to the design and light guide. Furthermore, the objective must exhibit precise field flattening characteristics for object measurement. These types of objectives, which are specially optimized for reflected light specimens, can be recognized by the abbreviation "EPI" (e.g., the ZEISS 50x EC EPIPLAN # 422070-9961-000). Among other things, special antireflection coatings reduce the reflections from the object and the optical calculation does not provide for a cover glass. The digital camera requirements for metallic samples must be optimized for measuring and documentation purposes. A highly dynamic imaging camera chip displays metal surfaces and their high levels of contrast in an ideal way. Special industrial software packages (such as "ZEN core" and its material modules) are available to provide assistance in using a camera of this type (for example, the ZEISS Axiocam 305) in practical settings. Microscopes for beginners, such as the "ZEISS Primotech," include the most common contrasting techniques and can also be used with simplified "MATSCOPE" software and tablet solutions ("MATSCOPE" for iPads).

The rapid optical development of the "digital microscope" is also making it an increasingly interesting tool for structural analyses. These devices are becoming increasingly significant thanks to their ease of use and their combination of the advantages of stereomicroscopy with those of reflected light microscopy. They therefore also cover a relatively broad magnification and application range. In particular, reflected-light microscopes do not offer the same opportunity to digitally post-process images with a wide range of measuring tasks. However, digital microscopes are unable to keep pace with the good resolution offered by reflected-light microscopes, which is a drawback when working with very small structural elements (cf. also Figs. 3 and 4).

Fig. 20: *Versions of light microscopes for structural analyses. In addition to inverted and upright material microscopes (such as the A ZEISS Axiovert.A1 MAT and ZEISS AxioLab.A1 MAT), digital microscopes such as the ZEISS Smartzoom 5 are increasingly becoming an alternative.*

Please note: Special optics must be used, as the excitation and object lighting are directed through the same optical system. These are denoted by the abbreviation "EPI."
Cameras must be capable of processing high contrast values.

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