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Lab Course Notes



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## **Metallography Lab Course**

# Institute for Applied Materials – Materials Science and Engineering

The lecture notes are partially derived from older versions created by former members of IAM-WK.

## **Table of Contents**

| 1                           | Obje | ectives of Metallographic Analyses     | 1   |
|-----------------------------|------|--|-----|
| 2                           | Stru | icture and Microstructure              | 1   |
| 3 Outline of the Lab Course |      |  | 2   |
|                             | 3.1  | Sample Extraction                      | 2   |
|                             | 3.2  | Mounting                               | 3   |
|                             | 3.3  | Grinding                               | 3   |
|                             | 3.4  | Polishing                              | 4   |
|                             | 3.5  | Contrasting                            | 5   |
|                             | 3.6  | Imaging                                | 6   |
|                             | 3.7  | Qualitative and Quantitative Analyses  | .11 |
|                             | 3.8  | Documentation                          | .14 |
| 4                           | Rele | evant Fundamentals from Other Lectures | .14 |
| 5                           | Lite | rature                                 | 15  |

## 1 Objectives of Metallographic Analyses

The major objective of metallography is the *qualitative and quantitative examination of the microstructure* of metallic materials. By correlation of these investigations to the technological and scientific conditions of materials manufacturing and processing and to structural information obtained by materials analytics, a holistic understanding of (crystal) structure-microstructure-property relationships can be derived. In most cases, methods of *advanced materials analytics/characterization* cannot be unequivocally distinguished from metallographic procedures as information of both, (crystal) structure and microstructure are needed and/or obtained by these methods.

Metallographic methods were more or less directly transferred to other materials classes, like polymers and ceramics, leading to the term *materialography*. The lab course will provide overview about classical and modern metallographic procedures that might be applied in the same or similar way also in materialography.

## 2 Structure and Microstructure

Structure and microstructure determine mechanical and functional properties of materials, and so metals and alloys. While crystal structure is defined in most cases by the alloy choice, microstructure can be deliberately tailored in order to achieve specific properties.

*Structure* in the sense of the present lab course is defined by the atomic mockup of a material. When the material is crystalline, crystal structure might be more precise to differentiate against microstructure. Elastic properties of metals and alloys are for example mostly determined by the crystal structure of the material and to less extent by the microstructure.

*Microstructure* in contrast covers the description of all constituting phases and defects including information about their type, shape, size, orientation and distribution. Within this description, a phase displays structurally and chemically homogeneous regions, the properties of which change continuously when thermodynamic state variables are changed. In contrast to phases, microstructural regions/constituents can be differentiated as their properties, like morphology (e.g. type, shape, size, orientation and distribution) allow to reconstruct their formation mechanism. Microstructural regions can thus be multiphase, like pearlite (eutectoid regions in steels) and ledeburite (eutectic regions in cast iron). Alternatively, different microstructural regions can belong to one and the same (thermodynamic) phase, like primary (formed from the liquid), secondary (formed from supersaturated austenite) and tertiary (formed from supersaturated ferrite) cementite. All properties involving plastic deformation phenomena (for example strength, ductility, toughness, etc.) are primarily determined by the microstructure of metals and alloys.

There are multiple definitions of structure and microstructure available in the field which partially overlap and might not allow a strict classification of a feature to either of the two categories. The above-mentioned definitions are chosen for the present lab course due to their simplicity and a rather convenient way of application. However, adoption might be needed when the methods and tasks of the lab course are transferred to other subjects or fields.

## 3 Outline of the Lab Course

The metallographic preparation needs to fulfill the following requirements to obtain the goals mentioned above:

- Relevance and representativeness of the microstructure information
- Reproducibility of the preparation
- Unaltered microstructural information
- Documentation

These aspects are relevant to all preparation steps which are typically:

- Sample extraction
- Embedding
- Grinding
- Polishing
- Contrasting
- Imaging
- Qualitative or quantitative Analyses
- Documentation

#### 3.1 Sample Extraction

Imaging of microstructures typically requires magnification as the microstructural constituents are small compared to the size of a component or semi-finished part. Thus, it is necessary to cut samples from larger pieces for the investigations. Relevant specimens for metallography are typically in the size of several mm<sup>3</sup> up to cm<sup>3</sup>.

As these samples are much smaller compared to the pieces they were taken from, the selection of the *position* of the sample is important for the relevance of the findings on these smaller samples. In case of damage analysis for example, extraction of samples close to failure regions but also from otherwise unaltered regions can be useful to reveal the origin of damage. The extraction position needs to be carefully documented.

Secondly, the orientation of the sample within the original part is important and needs to be documented throughout the following steps. Different types of sections are usually taken, e.g. cross sections or longitudinal sections. The cuts are usually performed perpendicular to one of the principal sample directions. In certain cases, flat angles might be used to create a better visibility of a feature in the section. Different types of sections and cuts are visualized in Figure 1 and Figure 2.







Figure 2: Schematical comparison of normal (left) and inclined cut (right) on a thin film to increase the section through the film in the image according to Ref. [1].

As all other preparation steps, sample extraction should not lead to microstructural changes. Depending on the extraction method, following changes might occur in the microstructure close to the cut: *mechanical, thermal* and *chemical*. Frequently applied extraction methods that restrict microstructural changes are mechanical with cooling (for example high speed cut-off grinding), electro-chemical or electrical erosive (for example electrical discharge machining).

#### 3.2 Mounting

In order to handle the small samples extracted from the larger pieces and to ensure homogeneous preparation to the edges of the samples, the specimens are usually mounted in resin. There are three different categories of mounting:

*Hot (compression) mounting*: Thermosets or thermoplastic materials can be used to mount the specimen. Mounting is then performed in a compression mounting press at elevated temperature and under uniaxial compression stress. The microstructure needs to be stable against these conditions. Due to high hardness (for example by filling the thermoset with ceramic particles) and utilization of a press, hot mounting ensures high edge sharpness and accurate shape of the mounted sample.

*Cold, castable mounting*: Epoxy, polyester resin or methyl methacrylate can be used in liquid form to cast the mount. Most mounting substances come as two-component raw materials and are mixed short before casting. The heat of reaction can lead to substantial temperature increase during mounting, especially when curing occurs within short time.

*Infiltration*: Specimens with pores or cracks might be infiltrated to avoid further damage of the material during preparation. Curing of the mounting material is then performed under vacuum or pressure.

### 3.3 Grinding

After mounting, the samples need to be ground to a flat surface in order to allow imaging. During the following grinding steps, the surface roughness is stepwise reduced. This is performed by grinding with consecutive reducing the grain size of the grinding paper, for example P120 down to P4000. "Grit" labels grinding paper according to ASTM while "P" designates grits according to the European FEPA standard. It is important to ensure sufficient cooling during the grinding steps to avoid intense mechanical, thermal and chemical interaction of the grinding paper (abrasive and substrate) with the material to grind. Furthermore, the cooling water removes wear particles from the process. Typical abrasives are SiC or diamond. In case of a target preparation, an anticipated depth is achieved during the grinding process by utilizing special sample holders. Lapping is an alternative to grinding and sometimes utilized. The method differentiates with respect to the type of adhesion of the abrasive to the substrate and the substrate itself. The differences are visualized in Figure 3.



Figure 3: Schematic on the differences of grinding (left) and lapping (right) according to Ref. [1].

#### 3.4 Polishing

Subsequent to grinding (usually P4000 equivalent to an abrasive grain size of approximately 5  $\mu$ m), surface finish is often not sufficient to allow for high quality microstructural imaging. Surface roughness and interaction layers by the previous preparation steps remain too dominant. Thus, both, roughness and surface layers need to be further reduced by the following steps. There are several polishing methods available.

*Mechanical polishing*: Mechanical polishing is performed on rotating disks equipped with textile fibers. These cloths are of different materials, weave types and of different thicknesses depending on the material to polish. Polishing suspensions that contain the abrasives are available with diamond (typical sizes of 30 down to 0.25 or even 0.05  $\mu$ m) or alumina (sizes of 1 to typically 0.25  $\mu$ m).

*Chemical or mechanical-chemical polishing*: Especially for very soft materials, like pure metals which tend to smear or to build up thick interaction layers, might be polished chemically or mechano-chemically. Chemical polishing is performed without macroscopic current and linear surface removal. Removal is controlled by dwell time and etchant concentration and temperature. A combination with mechanical polishing is useful.

*Electro-polishing*: A ground surface is used as anode and submerged in an electrolyte. The cathode is usually made of the material to polish, Pt or stainless steel. Polishing conditions are achieved when the surface of the sample is anodic removed. In contrast to mechanical polishing, polishing times are usually much shorter and by definition, no additional plastic deformation is involved in the process. However, the number of parameters to adjust is larger and uneven/phase-specific polishing might occur in the case of multi-phase materials.



Figure 4: Schematic representation of an electro-polishing device (left) and schematical current density vs. potential curves according to Ref. [1].

#### 3.5 Contrasting

Without contrasting, polished surfaces would hardly show any contrast due to their mirror-like finish. Hence, microstructural information needs to be created by contrasting the surface. Following techniques are frequently applied:

*Electro-chemical methods*: Similar to chemical polishing, electro-chemical etching utilizes surface removal without macroscopic currents. Under etching conditions, the removal is site-specific which creates a characteristic surface topography to be imaged by a proper technique. The chemical reactions are reduction-oxidation reactions. Elements located prior to H in the galvanic series are etched by acids under the development of H<sub>2</sub>. Elements with galvanic potentials higher than H are etched by acids only when additional oxidizers are added. Slightly different potentials on the sample surface lead to local element formation with cathodic and anodic regions, for example by (i) different phases (see Figure 5), (ii) different orientations, (iii) deformed and undeformed regions, etc. Depending on the type of localization, (i) grain boundary etching or (ii) grain facet etching occurs (see Figure 6), (iii) orientation-depending film formation (chemical deposition), or (iv) orientation-depending micro roughness can occur. In general, all relevant effects occur at the same time in different amount. Most etchants were empirically found over time and are documented in specialized literature. Several techniques have established to perform the etching: (i) submerging (static submerging), (ii) washing (etchant is flowing over the sample surface), (iii) rubbing or (iv) etch-polishing.



Figure 5: Schematic representation of the phase specific etching of pearlite with Nital (nitric acid  $(HNO_3)$  and ethyl alcohol  $C_2H_5OH$ ) according to Ref. [1]. C and A denote cathodic and anodic regions forming local elements.



Figure 6: Schematic representation of possible etching effects at grain boundaries (left) or surface facets of grains (right) according to Ref. [1]. C and A denote cathode and anode.

Similar to electro-polishing, the same apparatus can be used to etch the sample, see Figure 4. The etching potential is usually about 1/10 of the polishing potential.

*Physical methods*: Thermal etching can be applied to thermally resistant materials like ceramics. By a heat treatment at very high temperatures under vacuum, the surface tensions acting on grain boundary-surface junctions cause a diffusion flux of atoms from the grain boundary regions to the grain centers on the surface. By the fast surface diffusion flux, the equilibrium angle of the surface and grain boundary tensions is achieved and a small groove at the position of the grain boundary is formed, see Figure 7. Ion etching utilizes the selective surface removal by ion bombardment depending on the orientation of a grain or phase with respect to the incident beam. The selective deposition of thin films by physical methods can be used for various types of interference contrast.



Figure 7: Schematical representation of a polished surface with grain boundary surface junctions (upper part) and after heat treatment and the formation of grain boundary grooves (lower part) according to Ref. [1].

#### 3.6 Imaging

To image the contrasted samples, various types of radiation are used. Apart from optical microscopy using visible or invisible as well as polychromatic and monochromatic light, electron, ion and X-ray microscopy have also been established in various forms. Classical metallography often involves macroscope (visible, polychromatic light; stereo-type, low magnification microscope), light optical microscope (visible, polychromatic light; medium magnification; LOM) as well as confocal microscope. Electron beams are utilized in scanning electron microscopes (SEM) and transmission electron microscopes (TEM). Other types of common microscopes are atomic force microscopes (AFM) (no radiation used at all). Further relevant useful microstructural information is obtained by materials characterization methods, like X-ray diffraction (XRD).

Imaging techniques differentiate according to the type of image information that is often:

topographical

- chemical
- physical

Each of the information types are associated with a signal resolution and a lateral resolution.

The lateral resolution of an imaging technique is always restricted. However, the reasons for the resolution limits can be quite different. In general, lateral resolution might be described by the smallest distance *d* of two just individually resolvable features (points, lines, lattices) in an image. In case of light optical microscopes and imaging of bright and dark features for example, diffraction can limit the resolution. In these cases, the so-called Abbe limit can be used to estimate the lateral resolution limit:

$$d = \frac{\lambda}{n \sin \alpha} \tag{1}$$

with  $\lambda$  being the wavelength of the light, *n* the refraction index of the medium and  $\alpha$  the halve of the opening angle of the incoming beam.  $n \sin \alpha$  is referred to as numerical aperture of the microscope. As a rough estimate, *d* can be approximated as halve of the wavelength of the radiation used. Accordingly, polychromatic, white light in the case of LOM results in a resolution limit of several hundred nanometers up to 1 µm. If higher resolution is needed, LOM is not a proper choice.

The aforementioned considerations on diffraction limitation are ideal conditions which are rarely obtained under practical illumination and imaging conditions. In many cases, like LOM or TEM imperfections of the optical system cause resolution limits larger than expected just by diffraction effects. Relevant imperfections are spherical aberration, chromatic aberration, etc. Reduction of these imperfection is usually accompanied by significantly increasing effort and costs. In case of SEM, the resolution limit of the method is (by far) not related to these aspects but related to the interaction and information volume of the incoming radiation and the produced interaction products.

#### 3.6.1 Light Optical Microscopy (LOM)

Figure 8 depicts the schematical mockup of an LOM. The primary image is produced by imaging the object through the objective lens. Secondly, the primary image is projected via the eye piece to the human eye or without eye piece to a camera/imaging device. The magnification is obtained by the multiplication of the individual magnifications. Nowadays, LOM images are almost exclusively captured using digital imaging systems. Thus, magnification relating to certain standard sizes of photographs becomes less and less relevant. It is rather relevant to present width of field or micron scale bars for calibration.



Figure 8: Schematical mockup and optical beam path in an LOM according to Ref. [1].

Imaging is resolution limited by systematic flaws of the optical system in many cases. *Chromatic aberration* results from the different refraction indices of the lens materials as a function of the wavelength. Thus, the foci of individual wavelengths in polychromatic beams are different. Varying refraction depending on the distance to the optical center axis of a lens is called *spherical aberration*. It is corrected in lens design by using expensive aspherical lens designs. *Astigmatism* is caused by varying lens curvature across the lens surface. *Coma* results in imperfect imaging of off-axis point sources. Finally, *distortion* is another kind imperfection (worse lateral resolution when digitally corrected) of the optical system that causes a non-rectilinear image. All imperfections can be reduced by lens design, special sorts of glasses or coatings. However, costs are increasing tremendously and some imperfection will remain as a result of compromises.

Illumination is another aspect of LOM that needs attention with respect to homogeneity and high intensity but low scattering. Halogen bulbs (12 V, 25-150 W), Xe high pressure lamps (75-6500 W) or discharge lamps (Na: 589 nm, Th: 535 nm) are commonly used. The thermal radiation by the illumination might affect the lens system/microscope or the sample.

Two different imaging modes are to be considered. In bright field (LOM-BF, see Figure 9 left) illumination occurs almost perpendicular to the sample surface. Regularly reflected light is obtained from the flat sample surface and imperfections appear dark. In dark field (LOM-DF, see Figure 9 right) illumination is inclined with respect to the sample surface and regularly reflected beams are not imaged. Instead, imperfections diffract into the imaging beam path and become visible in an otherwise dark image.



Figure 9: Schematical representation of LOM-BF (left) and LOM-DF (right) according to Ref. [1].

Using polarized light, non-cubic substances or films (prepared during contrasting) can be imaged by the changing polarization of reflected light (for example FeS, AlN,  $Al_2O_3$ ). In the case of differential interference contrast, height differences on the surface create a phase shift of incoming and reflected beam. Interference of the beams results in contrast depending on the surface features. Other types of interference contrast are also utilized in various forms. Stereo microscopes are used for pseudo 3D imaging of samples.

#### 3.6.2 Scanning Electron Microscopy (SEM)

SEM provides a possibility to obtain high resolution images with a large variety of information carriers, including element-specific analysis using *energy dispersive X-ray spectroscopy* (EDS) or crystallographic analysis using *electron backscatter diffraction* (EBSD).

The flat sample is scanned by using a focused electron beam (usually  $\leq$  35 keV) and the resulting interaction products are acquired to form an image from the scanning process.

The SEM consists of an electron source, the electron column with electro-magnetic or electrostatic lenses, a vacuum sample chamber and the detector systems. The electron column is usually pumped to  $10^{-7}$  to below  $10^{-9}$  mbar. The sample chamber is kept at below  $10^{-5}$  mbar. Some SEM allow higher pressure to examine biological samples or to reduce charging of the sample. Navigation in the chamber is often supported by a video camera. The sample is manipulated using a multi-axes stage, the tilt axes of which are important for materials analytics methods like EBSD. Some SEM are combined with an extra column for *focused ion beam* (FIB) to locally mill the sample (often Ga or Xe ions) or deposit material (often C or Pt). The ions can also be used for imaging.

The detectors are attached at different angles with respect to the primary beam and the sample surface. The most relevant for materials science and engineering are: (i) The *secondary electron detector* (SE) is slightly above the sample or within the electron column ("in-lens"). (ii) The *backscattered electron detector* (BSE) is attached to the pole piece above the sample. (iii) The EDS detector is also located slightly above the sample with a certain tilt angle against the primary electron beam. (iv) The EBSD camera is attached almost perpendicularly to the primary beam.The sample needs to be tilted against the primary beam by 60 to 70°.

Electrons are emitted from the electron source, e.g. a W wire, an LaB<sub>6</sub> crystal or a field emission gun, and accelerated by an electrical high tension. *15 to 20 kV* are typically used, in rare analytical cases up to 30 kV might be necessary. On non-conducting materials or biological sample electrical high tension can be as low as some 100 V.

By using accelerated electrons, a resolution according to equation no. (1) below 1 nm might be expected. However, the extended *interaction/excitation volume* of the electrons in the sample restrict the resolution significantly. Figure 10 shows the region in which the primary electrons are decelerated in the sample by various elementary processes. Depending on the information carriers that are used to form the image, an *information volume* (often referred to as probe or probe volume) is defined form which the respective interaction products can be obtained. Note that all interaction products are formed in the entire interaction volume but the detection of the interaction products is only possible from a smaller region depending on their type and energy as they need to escape the sample for detection. In the case of TEM, most of the interaction volume is absent as thin foils are used – thus, the resolution is much higher and similar to LOM mostly restricted by imperfections of the optical system. The latest methods of electron microscopy combine the TEM method with a scanning focused beam.



Figure 10: Schematical representation of excitation volume and information volume for different interaction products of electron with the sample in an SEM according to Ref. [2].

In general, the excitation and thus information volumes increase when the acceleration voltage is increased or the average *Z* number of the sample is low. Lateral resolution might be improved by lower acceleration voltage when the signal intensity and excitation of the sample are sufficient.



Figure 11: Schematic representation of the changing excitation and information volumes for high (left) and low EHT (right) as well as low (left) and high *Z* (right) number according to Ref. [2].

In the order of relevance, the following investigations are often performed:

(i) SE are used to image the topography of the sample. SE exhibit a rather low energy of below 50 eV and, thus, the information depth is restricted only 1 to 10 nm. SE originate from inelastic interaction of the primary electrons with the electrons of valence and conduction band of the atoms. Due to the low information depth, a resolution *down to 1.5 nm* can be achieved.

(ii) In order to obtain Z contrast, BSE are used. BSE are quasi-elastically scattered electron with energies between 50 eV and the energy of the primary electrons. BSE are detected from up to 1  $\mu$ m depth depending on acceleration voltage and average Z number; thus, lateral resolution depends on these two aspects. As the backscattering coefficient significantly depends on the average Z number in the probe, a chemical contrast is depicted in BSE images. Additionally, electron channeling can be used to image crystal orientations and their variation. Rarely, a specific detector configuration can be used to image topography.

(iii) Apart from Bremsstrahlung, characteristic X-rays from the elements excited by the primary electrons can be used to assess the local chemistry of the sample. EDS or *wavelength-dispersive X-ray spectroscopy* (WDS) detectors are used in these cases. In ordert to quantify the chemical composition by evaluation of the peak intensities in the acquired X-ray spectra, corrections regarding Z number, absorption and fluorescence needs to be done. As X-rays are less absorbed than SE and BSE, lateral resolution of EDS/WDS maps is much worse compared to the other information carriers.

(iv) When the metallographic procedure results in deformation-lean surfaces, some of the BSE are diffracted by the lattice within the electron probe. The characteristic patterns can be imaged using an EBSD camera and evaluated regarding the crystal structure and its orientation with respect to the sample frame.

(v) Other techniques involve Auger electrons, cathodo-luminescence, and sample current measurements.

In order to image the sample under defined conditions, the primary electrons need to be conducted away from the focus point. In case of non-conductive samples, conductive films C or Au

can be deposited before SEM examination or gas injection systems might be used to get rid of surface charge. SEM are initially aligned when starting the work; this includes focusing, centering of the apertures, and correction of astigmatism.

#### 3.7 Qualitative and Quantitative Analyses

Microstructural constituents/regions can be described according to their structure and chemistry but also with respect to their *shape, size, and distribution*. The geometry of individual, comparably perfect crystals, so-called grains, is described using the enclosing defects, e.g. grain boundaries, phase boundaries, etc.

#### 3.7.1 Stereology

In order to quantitatively assess the microstructural regions using 2D images of sections, stereological methods are used. Certain assumptions/knowledge about the 3D mockup are needed in this case.

Following notations are used:

- *V* denotes volumes
- *S* denotes surfaces extended in 3D
- A denotes areas in sections
- *L* denotes lines
- *P* denotes points
- *N* denotes numbers/counts
- Capital letters refer to quantities obtained from the entire image or section; small letters refer to single objects
- Index 0 denotes test regions
- Brackets indicate phases
- Bars indicate average numbers
- Index *X* denotes the division by *X*

Following relevant quantities might be used:

- $P_P(\beta) = \frac{P(\beta)}{P_0}$  relative number of points assigned to phase  $\beta$  with respect to the total number of points in the test region
- $L_A(\beta) = \frac{L(\beta)}{A_0}$  relative length of intersections in phase  $\beta$  with respect to the total line length
- $V_V(\beta) = \frac{V(\beta)}{V_0}$  denotes the volume fraction of the phase  $\beta$
- $A(\beta) = \sum a(\beta)$  denotes the sum of all areas of phase  $\beta$  in the section

The most relevant evaluation methods are areal, line and point analyses. The methods are depicted in the following figures. Depending on the actual microstructures, different types of line arrangements or test patterns are used.



Figure 12: Schematical diagram to illustrate the difference between  $L_L$  (left) and  $P_L$  (right) when using test lines according to Ref. [1].  $L_L$  denotes the length of the line segments is used in relation to the total length of the test lines.  $P_L$  denotes the number of intersection points with the total length of the test lines.



Figure 13: Schematic diagram to illustrate the meaning of  $P_P$  as the ratio of a certain number of points within one microstructural region compared to the total number of test points.

The areal analysis is usually performed when phases can be separated by electronic binning. Manual assessment is rare. Apart from the relative area ratio  $A_A(\beta)$  of a phase, the relative line length per test area  $L_A(\beta)$  and  $N_A(\beta)$  can be quantified. The line analysis uses relative line length per total test line length  $L_L(\beta)$  or relative point number per total line length  $P_L(\beta)$ . Point analysis evaluates the relative point number  $P_P$ . In all cases, the images need to be representative for the microstructure, with a sufficient number of features but also of sufficient resolution to differentiate the features.

From the above-mentioned quantities, other 3D properties of the microstructure can be estimated assuming isometry (homogeneous length scale of the microstructural constituents) and isotropy (no direction dependence of the microstructural features):

- Volume fraction:  $V_V = A_A = L_L = P_P$
- Specific interface area:  $S_V = \frac{4}{\pi} L_A = 2P_L$
- Average particle diameter:  $d = \frac{V_V}{N_L}$
- Average particle distance:  $l = \frac{1 V_V}{N_L} = \frac{4 (1 V_V)}{S_V}$

#### 3.7.2 Microhardness

*Hardness* describes the materials' resistance against indentation. Thus, it depends on the resistance against both elastic and plastic deformation as well as the materials' ability to plastically deform.

The hardness value is often calculated from the ratio of applied force and projected indentation area. In some cases (for example Rockwell hardness testing), indentation depth under load is used instead. The hardness is a materials' property. Due to the inhomogeneous stress state under the indenter, hardness values should only be compared when they are acquired under the same conditions. For certain engineering classes of alloys, estimates are available in the standards to roughly compare or transfer hardness values into hardness data from a different testing method or even strength values. These conversions are only estimates. Hardness cannot be used for component design.

The most important testing variants are:

- Brinell
- Vickers
- Rockwell

In all cases, the load is quasistatic applied without bumping. After loading, the load remains constant for a defined dwell time. For Brinell and Vickers, the projected indentation area after unloading is used, while Rockwell utilizes the indentation depth under load.

The Vickers testing method according to DIN EN ISO 6507 utilizes a diamond pyramid indenter with quadratic basal plane. Indentation dwell time is usually 10 to 15 s. The projected indentation area is determined from the average diagonal  $d = \frac{1}{2}(d_1 + d_2)$  and the hardness according to:

$$HV = 0,189\frac{F}{d^2} \tag{2}$$

using applied force F in N and d in mm. The units of the hardness value are usually omitted in order to avoid confusion with stress values. The hardness is strictly no stress or strength value. A schematic of the Vickers test method is shown in Figure 14.



Figure 14: Schematic of the Vickers method according to Ref. [3].

Depending on the applied load *F*, following test ranges are subdivided:

•  $F \ge 49.03$  N: Macrohardness, hardness

• 0.09807 N  $\leq$  *F* < 1.961 N: Microhardness

Microhardness is used to characterize individual phases or microstructural regions and, thus, carries microstructural information.

#### 3.8 Documentation

The assessment, structuring and connection of meta data receives increasing attention. Therefore, the entire metallographic process and the results need to be stored in proper digital form. Accordingly, documentation is not only analogous or electronic anymore but also considers interrelations during the acquisition. At KIT, various electronic lab notebooks are available covering the above-mentioned aspects, for example <u>kadi4mat</u> or <u>chemotion</u>.



Figure 15: Graphical representation of materials manufacturing, processing, metallography and analysis in kadi4mat. All datasets are structured and contain meta data and actual results and are connected in a consistent manner.

#### **4** Relevant Fundamentals from Other Lectures

In order to prepare for the lab course, revision of fundamentals on the following subjects are important:

- Crystal defects
- Fe-C and Fe-Fe<sub>3</sub>C phase diagram

- Time-temperature-transformation or continuous cooling transformation diagrams
- Microstructures in steels and cast iron (close-to-equilibrium, quenching, tempering, engineering heat treatments)
- Modification by alloying

Details are found in the lecture materials on

- Materials Science and Engineering 1/2 for students in mechanical engineering or
- Materials physics/Metals for students in materials science and engineering

Relevant literature references are also found in the other parts of the lab course materials.

#### 5 Literature

- [1] H. Schumann: Metallographie, Leipzig: Deutscher Verlag für Grundstoffindustrie (1991)
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- [3] E. Macherauch, H.-W. Zoch: Praktikum in Werkstoffkunde, Wiesbaden: Springer Fachmedien (2014)