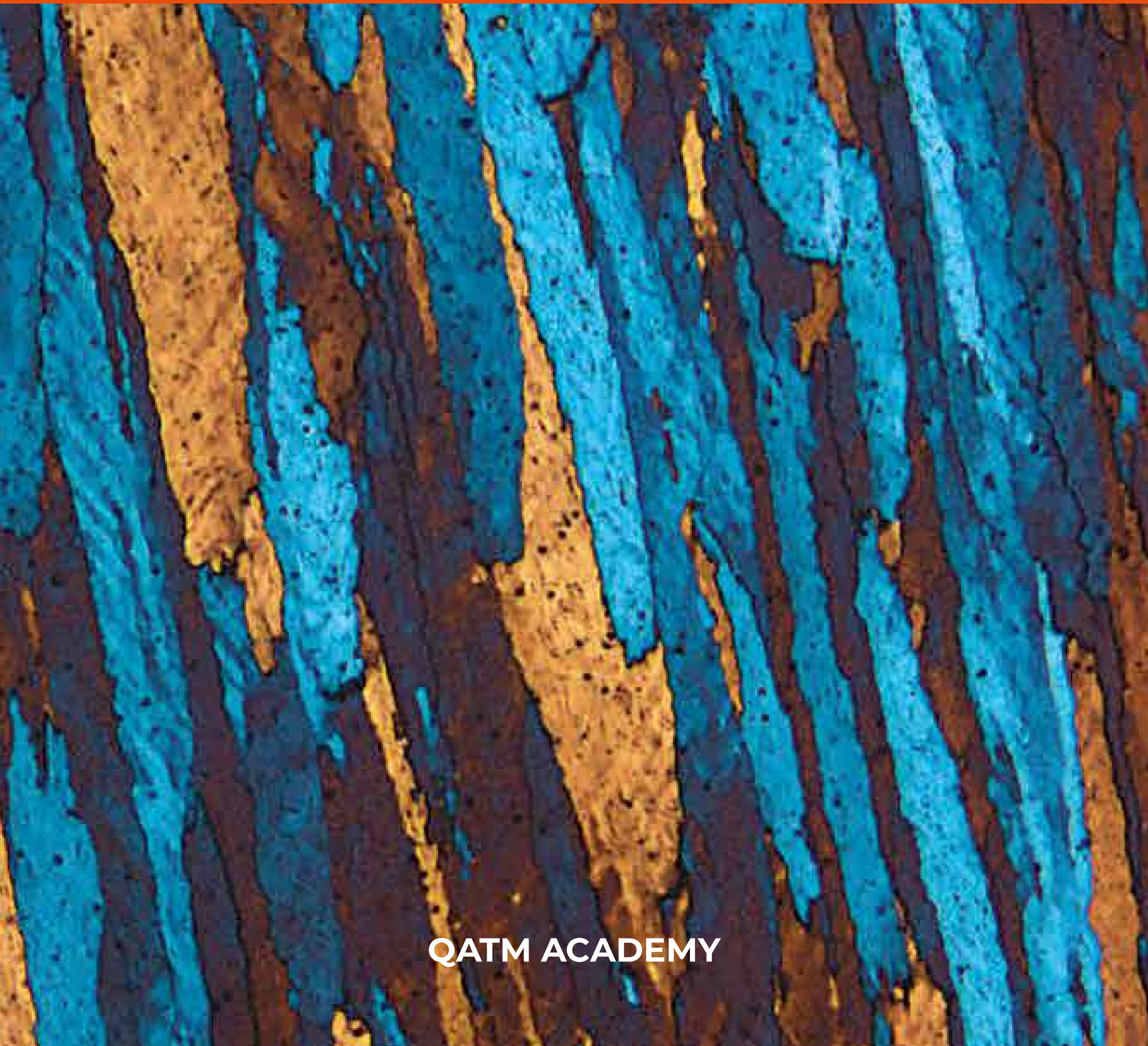
QUALITY ASSURED

Expert Guide

Materialography/Metallography



QATM ACADEMY

Expert Guide

Materialography/Metallography

- Guide for materialographers and beginners
- Tips & Tricks
- Artefacts avoidance
- From sampling to the finished cut

QATM AKADEMIE

INTRODUCTION

In order to be able to examine solid materials and/or materials macroscopically or microscopically, they have to be properly prepared using materialographic methods.

This book contains an introduction with a detailed description of the individual preparation steps. Solutions to problems as well as numerous other aids for application are described in detail.

The aim is to provide a comprehensive guide for all those who deal with this subject:

- By dealing with all topics and work steps in the sequence
- Through a detailed presentation of errors that occur frequently. This ensures the preparation of an artefact-free structure for subsequent analysis.
- A brief insight is given into the rules and physical and chemical laws that are at the background.

The systematic structure with comprehensively photographically documented examples is suitable for beginners as well as experienced users who want to learn or refer to a particular preparation technique.

With documented examples and a comprehensive selection of images, this Expert Guide is structured to support both the experienced materialographer and beginner alike.

WHAT DOES MATERIALOGRAPHY MEAN?

The term materialography used today is a factual extension of metallography, which was already brought to life with the age of microscopy and the first viewing of micrographs. In recent times, many other groups of materials, such as ceramics, plastics and composite materials are examined in the same way, which has expanded the original range of applications.

The aim is to describe the structure of the material qualitatively and quantitatively, i.e. the size, shape and distribution of the lattice structure defects and phase boundaries. This requires a suitable and defect free preparation of the sample.

The base for a materialographic sample preparation is in the field of physics and chemistry. The exact procedure depends on the material characteristic data, which must be known, as well as the properties, the composition and heat treatment condition.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1. Introduction to materialographic sample preparation

The path to evaluable macro and micro sections leads via materialographic sample preparation. The preparation is done by the following preparation steps:

- Removal by wet abrasive cutting
- Mounting
- Grinding
- Polishing
- Etching
- Analysis

This sequence of steps is not always completely necessary and not always correct for every examination, but nevertheless serves as a guideline for the materialographic sample preparation.

1.1 WET ABRASIVE CUTTING

The so-called wet abrasive cutting is a machine-based cutting process. In the field of materialographic sampling, this usually means the mechanical segmentation of components to be examined.

For this purpose, cut-off wheels of different thickness, structures and bonds are used on an appropriate wet abrasive cut-off machine. Normally, they consist of a synthetic resin or rubber bond of a certain hardness in which aluminium oxide or silicon carbide grains of a certain grain size are embedded. As a rule, the following principle applies:

> **For hard materials, a cut-off wheel with soft bond is recommended; for medium-hard materials, a cut-off wheel with medium-hard bond and for soft materials, a cut-off wheel with hard bond are used.**

Most manufacturers specify either a manufacturing or best-before date for their cut-off wheels. This must be observed since the epoxy resins of the bond are hygroscopic, i. e. they absorb water. Water in the bond can lead to embrittlement of the cut-off wheel after a few months or years. In this condition, the cut-off wheel is still usable, but with significantly higher wear and risk of breakage. For special materials, such as titanium or plastics, cut-off wheels with silicon carbide as abrasive grain (also resin-bonded) are generally used.

Diamond cut-off wheels are used for very hard materials (ceramics, glass fibre reinforced plastics, minerals, glass, etc.). There are two different bond types to choose from: Metal-bonded cut-off wheels, where the diamonds are usually embedded in a bronze bond, or those with a synthetic resin bond. The latter are particularly suitable for cutting very brittle materials. When the cut-off wheel is run in and out, the sample surface is prevented from breaking off, thus reducing the reworking that would be necessary to remove the damaged area.

1.1.1 SAMPLING (CUTTING)

The following requirements apply to sampling:

- Cutting by wet abrasive cut-off machine with sufficient cooling and rotating disc (e.g. no band saw). Otherwise, the deformation in the surface is so high that it cannot be rectified without reworking through tedious grinding.
- Optimum cooling is indispensable for evaluation. An excessively heated surface gives rise to misconceptions – artefacts simulate a false result.
- Corrosion protection agent must be added to the coolant, otherwise there is a risk of surface oxidation at the cutting surface. This is also important to protect the interior of the cutting device.
- The size and design of the cutter depend on the sample size – the criterion here is the so-called cutting capacity.

1.1.1.1 CUT-OFF WHEEL SELECTION

Wear (i.e. abrasion resistance), service life and cutting performance are decisive for the quality of the cut-off wheel. Abrasive agents (aluminium oxide, silicon carbide, diamond, cubic boron nitride (CBN), etc.) and bonding (metal, synthetic resin or rubber) are characteristics of the wheel composition.

1.1.1.2 AREA OF APPLICATION OF CUT-OFF WHEELS

- Corundum discs (aluminium oxide – Al_2O_3) with synthetic resin bond for all steels, depending on hardness
- Silicon carbide discs (SiC) for soft and medium-hard non-ferrous metals as well as hard non-metallic materials (glass, stone)
- Diamond cut-off wheels (C) for hard ceramic materials, ceramic composites and geological materials (rocks)
- Boron nitride wheels (CBN – Cubic Boron Nitride) for tough Co- and Ni-based alloys, hard metals, hard composite materials, and hard to very hard carbon steels.

> **Soft-bonded wheels are suitable for cutting harder materials, whereas hard-bonded wheels for cutting soft materials**

The grain size of the abrasive in the cut-off wheel is an aspect that must not be neglected when cutting materialographic samples. It should be between 45 and 180 µm. This allows an optimally cut surface, which should facilitate further preparation, provided all parameters are observed. A very fine grain size is recommended when using thin cut-off wheels. A fine grain size is also important if burr-free cutting is required.

The degree of hardness of a wet abrasive cut-off wheel does not say anything about the hardness of the abrasive grains, but rather about the hardness of the bond, i.e. about the resistance of the individual abrasive grain to breaking out of the bond material.

- A softer bond should be used for large-area contact points between the workpiece and the wet cut-off wheel. This also ensures that worn abrasive grains are broken out. This type of cut-off wheel is also well suited for heat-sensitive materials.
- In the case of smaller contact areas, a harder and more stable wet cut-off wheel is however suitable. Those are also used for cutting tubes and profiles. The advantage of this type of cut-off wheel is that it has less contact with the workpiece since the wall thicknesses is usually small.

The following images illustrate the possible errors when using wet cut-off wheels.

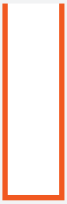
<table>
<tr><th colspan="3">THE CORRECTLY SELECTED CUT-OFF WHEEL WILL PRODUCE EDGE SHAPES LIKE:</th></tr>
<tr>
<td>In case of solid material, rounded edges indicate the desired result. The correct cut-off wheel was used in the correct manner:
</td>
<td>Sharp-edged corners are the sign of the correctly selected cut-off wheel for solid or profiled material as well as tubes of medium wall thickness:
</td>
<td>Concave corners are the expected result when cutting thin tubes and cross-sections:
</td>
</tr>
</table>

Figure 1: Edge shapes of cut-off wheels

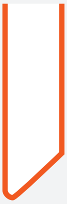
<table>
<tr><th colspan="3">INCORRECTLY SELECTED OR APPLIED, THE WET CUT-OFF WHEEL FORMS THE FOLLOWING EDGE SHAPES:</th></tr>
<tr>
<td>If the edges are sharp, an excessively hard cut-off wheel was used. The tapering effect can cause cut-off wheel breakage and burn marks:
</td>
<td>Bevelled edges indicate incorrect or insufficient cooling. This can cause the cut-off wheel to lateral run-out and shorten its service life:
</td>
<td>Glazed corners are formed if the abrasive grit is not broken out and the wheel may get clogged. In addition to an incorrectly selected wet cut-off wheel, excessively slow feed rate or insufficient rotational speed can also be the cause:
</td>
</tr>
</table>

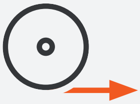
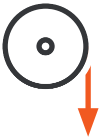
Figure 2: Edge shapes of cut-off wheels

1.1.1.3 WET ABRASIVE CUT-OFF MACHINES

In materialographic sample preparation, only wet abrasive cut-off machines are used for segmentation of workpieces, since this is the only way to avoid structural changes.

Optimum cooling is just as important for the processing result as precise cut-off wheel guidance. The cooling liquid should consist of a mixture of a boric and amino acid-free corrosion protection agent with water. An appropriate concentrate is diluted with water in a ratio of e.g. 1:30 (1 litre corrosion protection agent: 30 litres of water). The manufacturer's instructions are applicable.

Cleanliness (generally the top priority in materialographic sample preparation) must be ensured for the cutting chamber and drain system – this influences the cutting precision and the service life of the machine.

THE CUTTING PROCESS ITSELF IS DIVIDED INTO THE FOLLOWING PROCEDURES:	
	**TRAVERSE CUT** **Two versions are possible in this case:** 1. The sample table with the workpiece previously fixed on it is moved to the fixed cut-off wheel manually or automatically. 2. The cut-off wheel is moved accordingly.
	**CHOP CUT** The workpiece to be cut is firmly fixed on the cutting table. The cut-off wheel is then manually or automatically guided from top to bottom into the fixed workpiece.
	**STEP-CUT X** The workpiece is not cut in one cutting step, but in layers (stepwise). This type of cut is particularly suitable for cutting the large, thick workpieces of solid material.
	**STEP-CUT Y** The workpiece is not cut in one cutting step, but in layers (stepwise). This type of cut is particularly suitable for cutting the large, thick workpieces of solid material that must be clamped on edge (plates, angles, etc.).

3. Artefacts in materialographic sample preparation and their avoidance

This section contains a number of tips and tricks that can be very helpful for materialographic sample preparation. Errors, which the user can avoid easily, are presented as well.

3.1 CUTTING

3.1.1 SAMPLING THROUGH WET ABRASIVE CUTTING

The main error that the user must avoid during wet abrasive cutting is excessive heat generation, due to insufficient cooling. In this case, the generated heat causes the materials microstructure to be unpredictably altered in an impermissible way – it no longer represents the original material structure (figure 42).

This happened with the sample shown in figure 43 on the left side. The intensive tarnish colours are unmistakable, compared to the correctly cut sample in the figure on the right.

Figure 42: Incorrectly cut sample

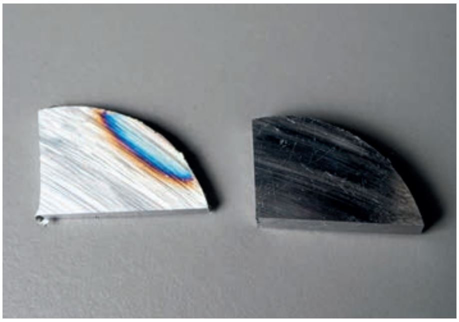


Figure 43: Sample pieces from incorrectly cut sample

The damaged sample can be clearly identified after preparation by the fact that the case-hardened area appears darker. It is the one that has been thermally damaged by improper cutting. The right sample was not affected by the cutting process.

A further comparison was made by small load hardness test in the carburised area of both samples. A significant difference in hardness was found.

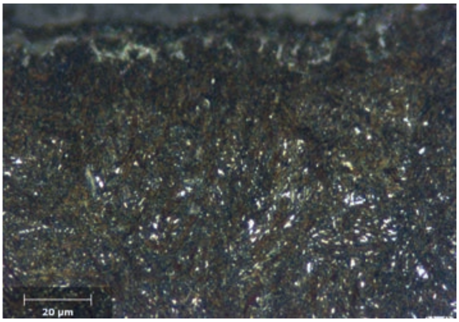


Figure 43.1: Structure of incorrectly cut sample

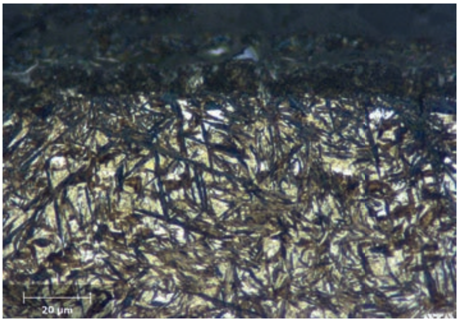


Figure 43.2: Structure of correctly cut sample

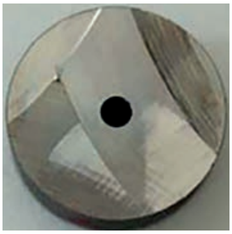
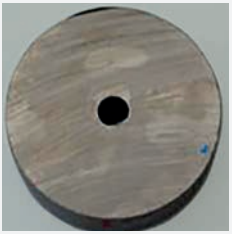
Incorrectly cut: 570 HV1
Correctly cut: 740 HV1

This means that the insufficiently cooled sample was tempered by the frictional heat. The microstructural changes to a less martensitic microstructure explain the decreased hardness values.

The situation is aggravated if the laboratory technician does not notice the faulty sample immediately. After cutting, both samples were embedded in an opaque hot mounting compound, so that the fault is no longer visible from the outside. Thus, an improper evaluation is very likely.

These artefacts can be prevented by selecting the correct cut-off wheel, the proper setting of coolant flow and a cutting feed rate adapted to the size of the part.

Other examples of errors:

PROBLEM	CAUSE	
MULTIPLE CUTTING APPROACHES	▪ Incorrect cut-off wheel ▪ Too small cutting wheel diameter ▪ Excessively high feed rate ▪ Poor cooling ▪ Inadequate clamping device	 Figure 43.4: Multiple cutting approaches
SURFACE HEATING		
DISCOLOURED MATERIAL SURFACE (STRUCTURE CHANGE)	▪ Incorrect cut-off wheel ▪ Excessively high feed rate ▪ Poor cooling	 Figure 43.5: Discoloured material surface
HEATING IN OUTLET AREA	▪ Poor cooling in the outlet area ▪ Excessively high feed rate when reducing the cross-section (use zone separation)	 Figure 43.6: Heating in the outlet area

3.1.2 SAMPLING THROUGH SAWING

It has already been mentioned that band saws have several disadvantages compared to wet abrasive cutting. This is illustrated here:

The sample produced by band sawing was cold mounted, ground, polished and then etched to visualize the microstructure. A strong deformation of the surface, caused by the saw teeth is characteristic. Therefore longer planar grinding times are necessary to remove the layer of plastic deformation.

4. Preparation suggestions and etching techniques

4.1 MACROSCOPIC ETCHING

4.1.1 THE SULPHUR PRINT ACCORDING TO BAUMANN

The sulphur print (Baumann print) provides information about the manganese-sulphide distribution (MnS) in the steel. Since this method of detection is no longer used so frequently and is partly forgotten, it is described in more detail here.

At present, a Mn:S ratio of 1.74:1 is aimed for in a steel melt. This ensures that all the sulphur contained is bound by manganese, the formation of iron sulphide (FeS) is prevented and the risk of red fracture is reduced. Red fracture is understood to be the cracking of the red-hot steel (800-1000 °C) during forming. The degenerated eutectic of iron and iron sulphide has a melting temperature of 985 °C. The low formability of the iron sulphide causes an intercrystalline fracture (along the grain boundaries).

Sample preparation:

The sample to be tested is ground to a grain size of P320; the sample must be free of grease and corrosion residues.

The procedure and description of the chemical reaction:

A photographic paper (hard silver bromide paper) is soaked in 5 % aqueous sulphuric acid (H_2SO_4) for 10 minutes.

This process takes place in daylight. The silver bromide (AgBr – light-sensitive salt) dissolved in the light-sensitive layer swells in combination with H_2SO_4. The photographic paper treated in this way is placed on the section surface of the prepared sample and rolled out with a rubber roller. It remains there for 3-5 minutes (depending on the sulphur content). The photographic paper must not move during this process.

Various chemical reactions take place:

On the ground surface

$MnS + H_2SO_4 \rightarrow H_2S + MnSO_4$

In the photographic paper

$2AgBr + H_2S \rightarrow Ag_2S + 2\,HBr$

Ag_2S shows up as a brown deposit on the photographic paper

After stripping and washing, the photographic paper is further treated in a fixing bath (sodium thiosulphate dissolved in water). The fixing time is approximately 30 minutes.

In the fixing bath

$2Na_2S_2O_3 + AgBr \rightarrow Na_3[(S_2O_3)_2Ag] + NaBr$

The thiosulphate dissolves the excess silver bromide from the light-sensitive layer and forms a complex salt with it. The resulting image is now retained.

At lower sulphur contents, pre-etching is necessary. This is done either with the etching process according to Heyn or a 3% alcoholic nitric acid (HNO_3).

For titanium or vanadium alloyed fine grain structural steels no reaction takes place, titanium sulphide and vanadium sulphide cannot be dissolved in H_2SO_4.

The Baumann print also does not respond in welded seams.
There are several reasons for this:

- There is an extremely low sulphur content in the welding electrodes.
- The melt (weld metal) solidifies very quickly, and the sulphur is finely distributed, i.e. there are no sulphur accumulations.

The Baumann print is particularly suitable for the examination of quenched and unquenched cast steel, cast steel, steel blocks and steel profiles.

When evaluating it, the pure (light) outer layer (speck layer) and the brown core zone with sulphur segregations must be distinguished. Only a qualitative assessment is possible; a quantitative statement cannot be made.

Figure 242: Rail cross-section – Baumann print

4.1.2 OBERHOFFER ETCHING

Oberhoffer etching is used to detect:

- Primary crystallisation
- Fibre orientation
- Sulphur segregation
- Phosphorus segregation

Sample preparation:

The preparation for the Oberhoffer etching is more complex, because the sample has to be prepared to the final polishing as in a microstructure analysis.

Mode of action:

The Oberhoffer etching is both a precipitation etching and a solution etching. The chemically less noble areas (e.g. phosphorus segregation) are covered with copper, while the segregation-free zones (i.e. zones rich in iron) are strongly attacked. The etching time is seconds to minutes.

Figure 243: Oberhoffer rail cross-section

The etching reagent can be purchased as a ready-made solution.


QATM
TU Plan Fluor
NA 0.30 / WD 23.5
10x HQ
QATM
TU Plan Fluor
NA 0.80 / WD 1.0
50x HQ
QATM

A change to the original aspect ratio of the image is not permitted. The reproduction magnification can be determined for the respective reproduction by direct comparison with the scale bar.

In original pictures and reproduction enlargements, the true size **must** be shown by a scale bar within the image. This avoids misunderstandings which can occur due to subsequent magnification or reduction.

The reproduction ratio must always be indicated in the notation '500:1' (not 500x or V = 500 or 500x).

The creation of digital images is highly dependent on the parameters of the image recording systems. Both the optical and the electronic components have a great influence. All digital image recording devices must be calibrated in x and y directions. You should use a certified standard. When saving, a lot of additional information can (and should) be saved, such as the lens used, the calibration value in length unit/pixel or the resolution of the digital camera. This information can be relevant for a clear interpretation of the images.

5.2 HARDNESS TEST

Hardness is defined as the resistance of one body to the penetration of another, harder body.

This definition gives rise to the possibility of hardness test. In principle, a defined indenter is pressed into a surface with a defined force. From the depth to which the indenter penetrates the surface, the resistance to penetration, i.e. the hardness, can be deduced.

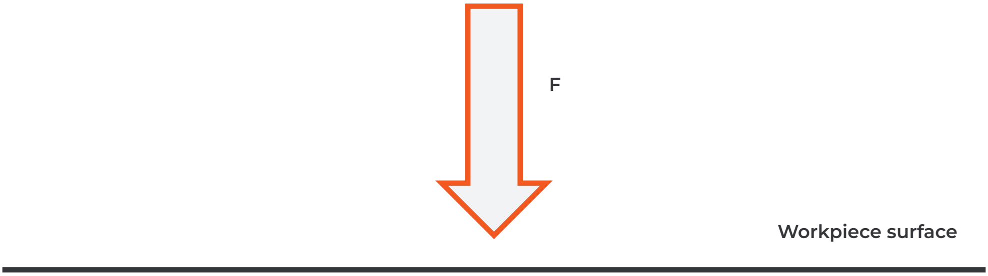


Figure 312: Definition of hardness test

In the course of time, various hardness testing methods have developed, but all of them are basically based on the principle described above.

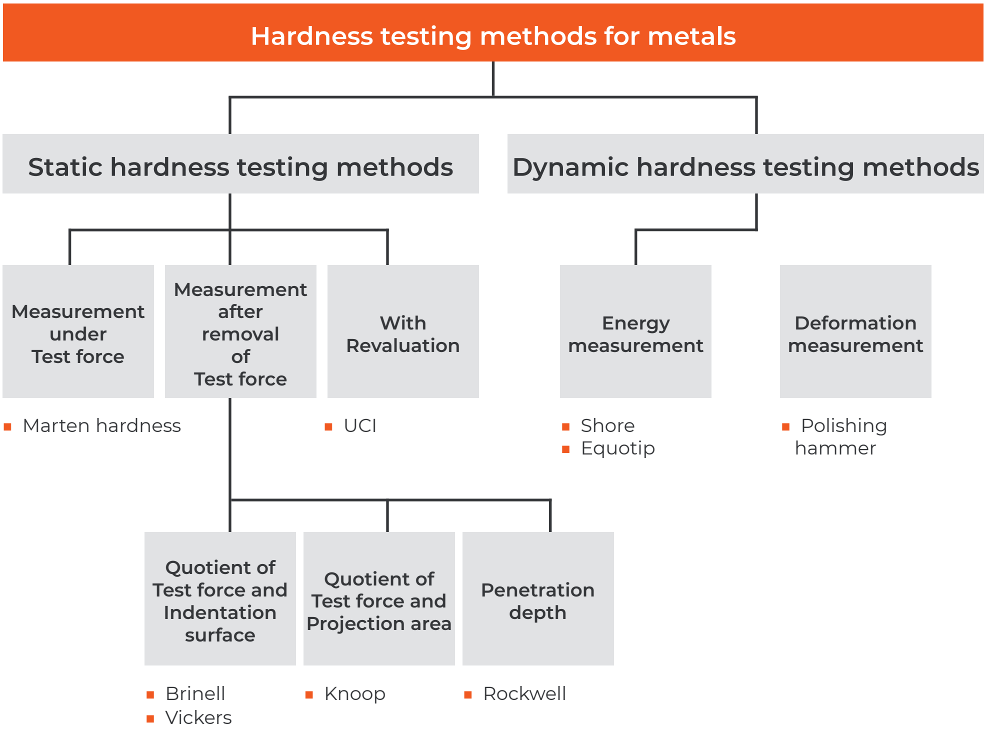


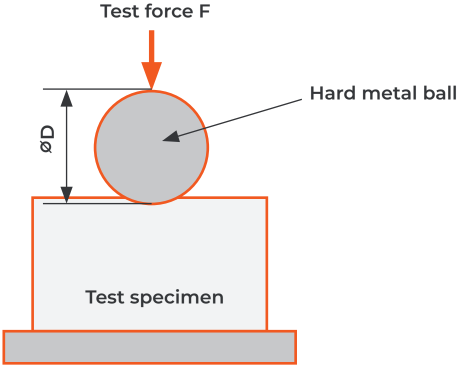
Figure 313: Hardness testing methods for metals

The four most common hardness testing methods are described below.

5.2.1 HARDNESS TEST ACCORDING TO BRINELL (DIN EN ISO 6506)

Johann August Brinell (Sweden) presented his new hardness testing method at the world exhibition in Paris in 1900.

In this method, a carbide ball is pressed vertically into the surface of a sample with a certain test force. This creates an indentation in the sample surface. The diameter of the indentation d is determined using a measuring microscope and the Brinell hardness is calculated or referred to from the corresponding tables.



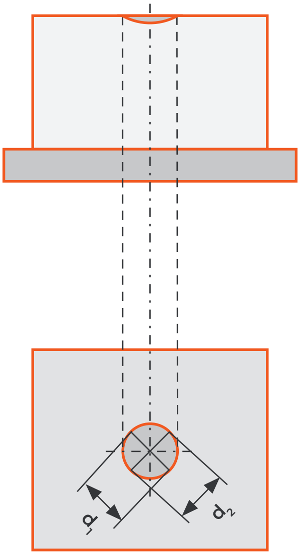


Figure 314: Fundamentals of Brinell hardness test

$$d = \frac{1}{2} * (d_1 + d_2)$$

$$HBW = 0.102 * \frac{F}{A} = 0.102 * \frac{2 * F}{\pi * D * (D - \sqrt{D^2 - d^2})}$$

Figure 315: Calculation formula for Brinell hardness test

For the Brinell hardness test, the selection of suitable test parameters is of decisive importance.

The Brinell method becomes inaccurate for very small or very large penetration depths. For this reason, the test load must be selected such that the following conditions are met for the mean indentation diameter:

$$0.24 * D < d < 0.6 * D$$

6. Safety in the materialographic laboratory

6.1 GENERAL INSTRUCTIONS

Although materialographic laboratories are relatively safe working environments, there is still a certain risk left, especially when handling chemicals. Laboratories must be designed and operated in accordance with the relevant regulations and the latest technological advancements. The specific activities in laboratories, especially those involving hazardous substances, require specific protective measures of a structural, technical, organisational, and personal nature.

At this point, an introductory overview of the measures necessary for safe working in laboratories is to be given. **This overview is not complete and expressly does not release the reader and/or user from researching the necessary information for his or her application in relevant local legal regulations and instructions.** Sources of information are, for example, the information materials published by professional associations and other institutions, such as the DGUV (German Social Accident Insurance).

Depending on the results of the risk assessments, only those measures, that are necessary to control or eliminate significant hazards, must be taken. In accordance with the order of priority of protective measures, the technical measures have priority over organisational and personal protective measures. **Due to the frequently changing manual activities in laboratories the use of a personalised saftey equipment (in particular safety goggles, laboratory coats, protective gloves) is necessary.**

Deviations from rules of technology are permissible if the same level of safety can be ensured by other means. This must be proven in each individual case.

A risk assessment for work in the laboratory usually results in certain generally required measures.

6.1.1 RISK ASSESSMENT

Since a large number of activities involving different hazardous substances are usually carried out in laboratories, the usual approach of determining protective measures on a case-by-case basis on the basis of the substance properties and the activities often cannot be applied. Safety in laboratories is determined by the construction, the equipment, the procedures, the operation, the devices as well as the qualification of the laboratory personnel. The combination of technical, organisational and personal measures minimises the hazards associated with laboratory activities. The construction and equipment of laboratories therefore essentially determine the activities that can be carried out in them.

Working in a fume cupboard is of particular importance in the laboratory, as the fume cupboard provides essential protection against the effects of physico-chemical properties, for example the formation of dangerous explosive atmospheres or the effects of moving parts and splintering materials, as well as against toxic hazards.

In laboratories, the following hazards from hazardous substances are typically to be expected:

- Risk of fire and explosion from flammable, liquid and gaseous substances
- Risk of damage to health due to solid, liquid and gaseous substances
- Danger from unknown, violent or continuous reactions
- Danger to eyes and skin from corrosive and irritant substances

In addition, employees are often endangered or exposed to other effects when working in laboratories:

- Inadequate or inappropriate lighting
- Unfavourable room climatic conditions
- Danger from containers with overpressure or vacuum
- Danger from hot or cold surfaces and media
- Noise from devices and systems
- Mechanical hazards from devices and systems

- Skin hazards due to wet work, especially when wearing gloves

- Risk of slipping due to wet floors, risk of tripping

- Repetitive strain injuries on the musculoskeletal system due to repetitive tasks or forced postures

- Mental stress due to repetitive tasks, time pressure, isolation, high demands on concentration

- Stresses on employees due to personal protective equipment

A risk assessment must take into account all aspects that can have a direct or indirect impact on safety. For example, the ergonomic aspect of lighting has a considerable influence on safety when working with hazardous substances. Poor visibility conditions – for example in an extraction unit – represent an increase in risk when working with hazardous substances.

Measures to protect against hazardous substances must be defined in such a way that, as far as possible, no additional hazards and stresses are caused by them.

It must also be taken into account that, in addition to activities involving hazardous substances, hazards may also arise due to special effects. These include, for example, hazards due to

- Ionising radiation
- Electromagnetic fields
- Optical radiation (laser, UV, IR)
- Biological agents

The risk assessment must therefore also check whether interactions of hazardous substances with these effects can occur during the activity, which can lead to an increase in danger (ignition of flammable air-vapour mixtures due to laser radiation).

The employer may only allow an activity involving hazardous substances to commence after a risk assessment has been carried out and the necessary protective measures have been taken.

The measures required for safe working in normal laboratory operation on the basis of the general risk assessment according to the Ordinance on Hazardous Substances can be regarded as given if the following general conditions are met:

- Construction and equipment in accordance with DGUV 213-850 (German Social Accident Insurance) and prevailing regulations
- Deployment of competent personnel
- Work in accordance with the relevant regulations and the latest technological advancement
- Work on a laboratory scale
- Work in accordance with DGUV 213-850 (German Social Accident Insurance)

If these general conditions are not met, a comprehensive 'risk assessment without comprehensive measures' according to No. 6 of TRGS 400 must be carried out in any case.

Further information can be found in the relevant regulations and in DGUV 213-850 (German Social Accident Insurance).

6.1.1.1 INFORMATION GATHERING

The source of information on the hazardous substances used is initially the labelling of the substances and the corresponding safety data sheet.

When keeping a list of hazardous substances, it is recommended not to exclude those hazardous substances whose use -at the moment- only leads to a low risk, as otherwise the list would also have to be adjusted immediately in case of changes or new uses. In addition, a list of hazardous substances, which advantageously takes the few laboratory chemicals that are not hazardous substances into account, provides a quick overview of stocks as well as procurement and disposal needs.

6.1.1.2 QUALIFICATION OF LABORATORY PERSONNEL

Laboratory personnel must be competent for the activities to be carried out. Expertise is determined by the type and duration of relevant training, general professional experience in the respective field and experience in the activities to be carried out.

In order to be able to examine solid materials and/or materials macroscopically or microscopically, they have to be properly prepared using materialographic methods.
This book contains an introduction with a detailed description of the individual preparation steps.
Solutions to problems as well as numerous other aids for application are described in detail.

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