Metallographic Sample Preparation Grinding and Polishing of Metals

Related terms
Grinding, Polishing, Metallographic sample preparation, Ductility

Method
Metallography is the art of preparing metallic samples by grinding, polishing and eventual etching for subsequent microscopic examination. Grinding and polishing is to prepare the specimen surface so as to enable the microstructure to be revealed by a suitable etching procedure.

Equipment
Grinding and polishing machine, 50-600rpm
Grinding platen, aluminium, dia. 200 mm
Polishing platen, PVC, dia. 200 mm
Splash guard
Lid
Magnetic foil, dia. 200 mm
Metal disk, dia. 200 mm
Polishing cloths METAPO-P, dia. 200 mm, for 10-6 μm diamond, pkg. of 10
Polishing cloths METAPO-B, dia. 200 mm, for 3-1 μm diamond, pkg. of 10
Polishing cloths METAPO-V, dia. 200 mm, for 1-0.1 μm diamond and oxide, 10
Final polishing cloths, MD-Nap, 200 mm, 5
SiC paper disks, d. 200 mm, grit 240, 100
Diamond suspension, 6 μm, 250 g
Diamond suspension, 3 μm, 250 g
Diamond suspension, 1 μm, 250 g
Diamond suspension, 0.25 μm, 250 g
Diamond stick P, 6 μm, 24 g
Aluminium oxide suspension, 0.05 μm, 1 l
Lubricant, water base, 1 l
Lubricant RED, 1 l
Coarse grinding disk, MD-Piano 220
Coarse grinding disk, MD-Primo 220
Fine grinding disk, MD-Piano 1200
Fine grinding disk, MD-Largo, 200 mm
Fine grinding disk, MD-Allegro, 200 mm
Set of eight raw samples consisting of:
- brass (CuZn40Pb2F44), copper (E-CuF25),
- aluminium (Al99zh), Al alloy (AlMgSiPbF28),
- steel (9s20k), alloyed steel (x12CrMoS17)
- brass, 600°C heat treated, steel, 750°C heat treated

Spray bottle, 500 ml
Cleaning and polishing tissues, pkg. of 50
Gloves, disposable, pkg. of 100
Wash bottle, plastic, 1000 ml
Dropping bottle, plastic, 50 ml
Funnel, d. 40 mm
Brush, universal
Labels, blank, 37x74 mm, 10 pcs.
Marking pencils, set, waterproof
Magnifier, 10x, dia. 25 mm

Recommended accessories:
- Metallurgical microscope, trinocular, magnification up to 400x
- Ultrasonic cleaning bath, RK100H
- Cleansing solution, concentrated, 1 kg

Fig. 1: Equipment for metallographic sample preparation
Task
1. Check the six metal specimens by means of the magnifier for any coarse defects.
2. Grind and polish the samples according to the general rules and the detailed instructions given below, considering the hardness and ductility data given in Table 1 and the basic processing guidelines specified in Table 2.
3. Evaluate the influence of the individual process parameters on the surface quality obtained in the intermediate steps and after the final polishing.
4. Try to optimize the grinding and polishing procedures.

Experimental Procedure and Results
As shown in Fig. 1, put the grinding and polishing machine on a clean laboratory table close to a sink and water tap. Take care that the environment is as dust-free as possible. Place the consumables and accessories beside the machine. If not used for some time, keep all auxiliary materials in closed boxes or bags to protect them against contamination by flying abrasive particles or dust.

The following general rules should be obeyed:
- Only utmost cleanliness will lead to a satisfactory final polish.
- Carefully wash your hands and clean the machine, disk and sample after each grinding and polishing step. It is recommended to wear disposable gloves in the polishing stages.
- An ultrasonic cleaner is a useful accessory for an efficient cleaning of the samples.
- Always use a polishing cloth only for one specific diamond grain size. In particular, avoid contamination of a polishing cloth used for final polishing, i.e. for 3 µm or 1 µm grain size, with 6 µm diamond grains. If this has happened, the cloth can then only be used for 6 µm polishing.
- When not in use, the cloths must be kept strictly separated. For storage, it is best to seal the dry cloths in plastic bags. Use a waterproof pen to label the metal backed cloth on the rear as well as the bag or box with the respective grain size.
- Use separate platens for grinding and polishing, e.g. the aluminium platen for grinding and the PVC platen for polishing.

![Recommended movement scheme of the sample on the grinding or polishing disk](image)

**Fig. 2:** Recommended movement scheme of the sample on the grinding or polishing disk

- It is recommended, especially in the grinding steps, to move the samples in circles of about 6 to 8 cm diameter, close to the periphery of the disk and usually counter to its direction of rotation, about half the time on the left and on the right side (see Fig. 2). This will help to wear the disk uniformly.
- For a gentler attack of the specimen surface due to a slower relative sample/disk speed, you may change the sample position to the central area of the disk and/or move the sample on the stationary disk for the last few seconds. This should be done with very slight or even no pressure.
- The pressure exerted with your hand on the sample, the speed of sample movement, the rotation speed of the disk, the kind of cloth, the abrasive and lubricant quantities, and the durations of the individual processing steps are important parameters that influence the result.
- All abrasion products must be removed from the grinding disks after each use by means of a brush and clean water and from the polishing cloths from time to time with distilled water and some household detergent.
- The wetted resin-bonded diamond grinding disk MD-Piano and the resin-bonded silicon carbide grinding disk MD-Primo should be dressed oc-

### Table 1: Sample materials (not heat treated), properties influencing the grinding and polishing behaviour

<table>
<thead>
<tr>
<th>Material/Code</th>
<th>Hardness (Vickers)</th>
<th>Ductility</th>
</tr>
</thead>
<tbody>
<tr>
<td>Brass CuZn40Pb2F44</td>
<td>~ 90</td>
<td>medium</td>
</tr>
<tr>
<td>Copper E-CuF25</td>
<td>~ 70</td>
<td>high</td>
</tr>
<tr>
<td>Aluminium Al992h</td>
<td>~ 25</td>
<td>high</td>
</tr>
<tr>
<td>Aluminium alloy AlMgSiPbF28</td>
<td>~ 60</td>
<td>medium</td>
</tr>
<tr>
<td>Steel 9s20k</td>
<td>~ 160</td>
<td>medium</td>
</tr>
<tr>
<td>Steel, alloyed x12CrMoS17</td>
<td>~ 240</td>
<td>medium</td>
</tr>
</tbody>
</table>
casionally by means of the included dressing stick, if the grinding efficiency seems to have deteriorated.

- The silicon carbide paper disks (grit 240) are designed for coarse grinding of very soft materials (e.g. aluminium) as an alternative to MD-Primo. Better results, however, may be obtained by fine grinding with MD-Piano 1200 as the first step. SiC paper should be used with plenty of water. An SiC paper grinding disk is consumed in most cases after a maximum service time of one minute.

- The lifetime of the grinding disks MD-Piano, MD-Primo, MD-Allegro and MD-Largo is very high, if they are carefully handled, and usually corresponds to the lifetime of more than 100 silicon carbide paper disks. They can be used until the grinding spots have been completely worn out.

- The aluminium oxide polishing suspension is to be used only in combination with aMETAPO-V or MD-Nap cloth for final polishing, particularly for soft metals. With hard materials, a satisfactory final polish is frequently already obtained after 3 μm diamond polishing. Always use a separate cloth for oxide polishing.

- The RED lubricant contains oily components and therefore provides a smoother attack.

- For exchange, cautiously lift the metal-backed disk from the machine platen and take care not to bend it too much. Permanently deformed disks must be discarded.

- After use, always cover the machine with the lid provided.

- It is advisable to use the 50 ml dropping bottles for economical dosage of abrasive suspensions and lubricants and for avoidance of environmental contamination by spraying. Provide the bottles with labels for identification.

<table>
<thead>
<tr>
<th>Material</th>
<th>Step 1</th>
<th>Step 2</th>
<th>Step 3</th>
<th>Step 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Brass</td>
<td>MD-Primo 220</td>
<td>MD-Largo/6µ DSt</td>
<td>METAPO-B/3µ DSu</td>
<td>METAPO-V/1µ DSu</td>
</tr>
<tr>
<td></td>
<td>Lubricant: water</td>
<td>Lubricant: w.b.</td>
<td>Lubricant: RED</td>
<td>Lubricant: RED</td>
</tr>
<tr>
<td></td>
<td>Time: 2 min</td>
<td>Time: 4 min</td>
<td>Time: 3 min</td>
<td>Time: 2 min</td>
</tr>
<tr>
<td></td>
<td>Speed: 300 rpm</td>
<td>Speed: 150 rpm</td>
<td>Speed: 150 rpm</td>
<td>Speed: 150 rpm</td>
</tr>
<tr>
<td>Copper</td>
<td>MD-Primo 220</td>
<td>MD-Largo/6µ DSt</td>
<td>METAPO-B/3µ DSu</td>
<td>METAPO-V/1µ DSu</td>
</tr>
<tr>
<td></td>
<td>Lubricant: water</td>
<td>Lubricant: w.b.</td>
<td>Lubricant: RED</td>
<td>Lubricant: RED</td>
</tr>
<tr>
<td></td>
<td>Time: 1 min</td>
<td>Time: 3 min</td>
<td>Time: 2 min</td>
<td>Time: 1.5 min</td>
</tr>
<tr>
<td></td>
<td>Speed: 300 rpm</td>
<td>Speed: 150 rpm</td>
<td>Speed: 150 rpm</td>
<td>Speed 150 rpm</td>
</tr>
<tr>
<td>Aluminium</td>
<td>MD-Piano 1200</td>
<td>METAPO-V/3µ DSu</td>
<td>MD-Nap/OSu</td>
<td>METAPO-V/OSu</td>
</tr>
<tr>
<td></td>
<td>Time: 1 min</td>
<td>Time: 3 min</td>
<td>Time: 1 min</td>
<td>Time: 1 min</td>
</tr>
<tr>
<td></td>
<td>Speed: 150 rpm</td>
<td>Speed: 100 rpm</td>
<td>Speed: 100 rpm</td>
<td>Speed: 100 rpm</td>
</tr>
<tr>
<td>Aluminium Alloy</td>
<td>MD-Primo 220</td>
<td>MD-Largo/6µ DSt</td>
<td>METAPO-V/3µ DSu</td>
<td>METAPO-V/OSu</td>
</tr>
<tr>
<td></td>
<td>Time: 1 min</td>
<td>Time: 3 min</td>
<td>Time: 2 min</td>
<td>Time: 1 min</td>
</tr>
<tr>
<td></td>
<td>Speed: 150 rpm</td>
<td>Speed: 150 rpm</td>
<td>Speed: 150 rpm</td>
<td>Speed: 100 rpm</td>
</tr>
<tr>
<td>Steel</td>
<td>MD-Piano 220</td>
<td>MD-Allegro/6µ DSt</td>
<td>METAPO-P/6µ DSu</td>
<td>METAPO-V/3µ DSu</td>
</tr>
<tr>
<td></td>
<td>Lubricant: water</td>
<td>Lubricant: w.b.</td>
<td>Lubricant: RED</td>
<td>Lubricant: RED</td>
</tr>
<tr>
<td></td>
<td>Time: 2 min</td>
<td>Time: 3 min</td>
<td>Time: 2 min</td>
<td>Time: 2 min</td>
</tr>
<tr>
<td></td>
<td>Speed: 300 rpm</td>
<td>Speed: 150 rpm</td>
<td>Speed: 150 rpm</td>
<td>Speed: 150 rpm</td>
</tr>
<tr>
<td>Steel, alloyed</td>
<td>MD-Piano 220</td>
<td>MD-Allegro/6µ DSt</td>
<td>METAPO-P/6µ DSu</td>
<td>METAPO-V/3µ DSu</td>
</tr>
<tr>
<td></td>
<td>Lubricant: water</td>
<td>Lubricant: w.b.</td>
<td>Lubricant: RED</td>
<td>Lubricant: RED</td>
</tr>
<tr>
<td></td>
<td>Time: 3 min</td>
<td>Time: 3 min</td>
<td>Time: 2 min</td>
<td>Time: 2 min</td>
</tr>
<tr>
<td></td>
<td>Speed: 300 rpm</td>
<td>Speed: 150 rpm</td>
<td>Speed: 150 rpm</td>
<td>Speed: 150 rpm</td>
</tr>
</tbody>
</table>

Table 2: Grinding and polishing guidelines

DSt = Diamond Stick, DSu = Diamond Suspension, OSu = Oxide Suspension, w.b. = water base
Now attach a magnetic foil to each of the platens (aluminium and PVC). Stick a METAPO-P cloth, METAPO-B cloth and METAPO-V cloth with their self-adhesive backs to three of the thin metal disks. The grinding disks and the MD-Nap polishing cloth are already provided with metallic backs for magnetic fixation. Attach the fresh water and drain hoses to the machine and connect the machine to the mains. Carefully read the Operating Instructions. Put the aluminium platen on the machine. Cautiously place a coarse grinding disk (MD-Piano or MD-Primo) on the magnetic cover of the aluminium platen, taking care not to bend it excessively.

Select a sample from the set, switch on the machine and set it at an operating speed of about 300 rpm or 150 rpm, depending on the sample selected (see Table 2). Apply some water from the spray bottle or lubricant from a dropping bottle on the rotating disk surface according to the specifications in the table. Hold the sample firmly with three fingers and push it gently with one of the circular faces down onto the rotating disk, moving it in circles of 6 to 8 cm in diameter at a speed of one to two full circles per second. Avoid too much pressure, which would produce deep scratches. Frequently change the position of the sample from one to the other side of the disk (see Fig. 2), with the last circles in the central region of the disk at minimum pressure. Lift the sample from the disk after some time, e.g. 30 seconds, to see if the circular grooves from the prior machine processing (turning lathe) have disappeared and have been replaced by a pattern of coarse scratches (cp. Fig. 3). If this is not the case, continue grinding with repeated inspection of the surface condition by means of the magnifier or an optional microscope. Keep the disk surface wet during the whole grinding procedure.

Clean the sample in running water and proceed to the next step. For step 2, replace the coarse grinding disk by a fine grinding disk MD-Largo or MD-Allegro, depending on the material. These disks require the addition of diamond abrasive. The diamond stick (6 µm grain size) is used to apply a small abrasive quantity on the disk surface by one or two light strokes across the running disk, from the centre to the periphery for uniform distribution. Use the recommended lubricant to keep the disk humid - not wet! - during the whole processing step. Inspect the surface repeatedly by means of the magnifier to see if all broad scratches from the previous step have been removed. After this step the surface should show a system of considerably finer scratches (cp. Fig. 4). Note the presence of three coarser scratches in Fig. 4, which may be remainders from step 1. Rinse the sample carefully in running water, clean the machine environment, and wash your hands to remove any residues of 6 micron diamond grains.

Now replace the aluminium platen by the PVC platen provided with a METAPO polishing cloth according to the information given in Table 2. Make the disk humid by putting one or two millilitres of the recommended lubricant on the running disk. Distribute some drops of the recommended polishing suspension from a drop bottle on the running disk, moving the bottle from the centre to the rim of the disk. Apply the lubricant and the diamond suspension very economically to keep the cloth uniformly humid, but avoid any surplus quantity to be splashed away from the cloth. Cloth polishing may require a somewhat higher pressure than that needed in the grinding steps. This polishing step will already produce a fairly good finish with only very fine scratches (see Figs. 5 and 6).

For the final step (step 4) the same strict rules of cleaning apply as in the previous steps. The diamond suspension is spread over the final polishing cloth like in step 3. For oxide polishing, a separate cloth METAPO-V or MD-Nap must be used, and no additional lubricant is to be applied together with the aluminium oxide suspension. Examples of possible sur-
face appearances after final polishing are shown in Figs. 7 and 8. If the polish is not yet fully acceptable after step 4 diamond polishing, a fifth step using a smaller grain size with MD-Nap may follow.

Fig. 5: Surface condition of aluminium alloy after step 3, magnification 200x

Fig. 6: Surface condition of brass sample after step 3, magnification 200x

Fig. 7: Surface condition of aluminium alloy after step 4, magnification 200x

Fig. 8: Surface condition of brass sample after step 4, magnification 200x

The aluminium sample requires a special treatment because of the extremely low hardness of the metal. Fine grinding on an MD-Largo disk would lead to the impression of many of the partially loose (rolling) diamond grains into the surface of the sample, leaving a condition as shown in Fig. 9. It is therefore recommended to apply a combined plane and fine grinding as a first step with a fine-grained MD-Piano 1200 with fixed diamond grains embedded in a resin matrix. The final processing is carried out on a very „soft“ cloth, having a high elasticity of the fibres, thereby largely preventing the diamond grains from being impressed into the soft aluminium surface (see Fig. 10).

Since the final results are highly dependent on the pressure forces applied to the specimen during grinding and polishing as well as on the relative sample/disk speed, the process would be fairly reproducible only if a repeatable, adjustable pressure could be applied. Therefore, in manual grinding and polishing, achieving a desired surface quality is a matter of „feeling“ and experience. Ideally, at the end of the final polishing step, any visible deformation (scratches and other surface defects) from the preceding steps should have been removed, and the sample should exhibit a mirror-like, virtually scratch-free surface.

Basic Theory
The behaviour of metals in mechanical surface treatment strongly depends on their macroscopic properties, i.e. hardness and ductility, which, in turn, are determined by their microstructures. A solid metal is normally produced by solidification from a melt. In the course of the solidifying process, micro-crystals are formed around incidentally produced nuclei, wherein the atoms arrange in an ordered three-dimensional system, called a crystal lat-
tice. The crystals grow in all directions until they touch their neighbours and form boundaries. Their final sizes, shapes and crystallographic orientations are random. Thus a macroscopic piece of metal has a microstructure consisting of many interlocking crystallites, also called ‘grains’. The microstructure determines the mechanical properties of the metal. It can be distorted by mechanical stress, e.g. processing by rolling or drawing, or by thermal treatments such as annealing, tempering and quenching. These processes alter the size, shape and/or orientation of the grains and thereby also modify the macroscopic properties, causing stress production or relief, ductility increase, hardening, etc. While in a pure metal the individual crystallites (grains) are normally equal in composition and structure (apart from elements forming polymorphic modifications), and differ only by size, shape and orientation, the microstructures of alloys are in most cases characterized by ‘phases’ of different composition (dual-phase or multi-phase structures). The formation of phases depends on the ability of the constituent metals to produce mixed crystals (complete or partial solid solutions) or intermetallic compounds. Complete solid-solution formers, e.g. gold and silver, form single-phase alloys. The composition of the phases, displayed in a ‘phase diagram’, is temperature and concentration dependent. The phase structure may have a very great influence on the physical and chemical properties of the alloy, e.g. melting range or melting point, corrosion resistance, electrical conductivity and thermal conductivity, expansion coefficient, tensile strength, abrasion resistance, hardness and ductility. This means that the microstructure of pure metals as well as the phase structure of alloys will also determine their behaviour in grinding and polishing processes.

The physical processes prevailing in grinding and polishing are determined not only by the mechanical properties of the material but also, apart from the operational parameters of the grinding and polishing procedures, by the inherent properties of the grinding disks, polishing cloths, abrasive media, and lubricants.

In principle, grinding and polishing are chip forming processes. Due to their hardness, their sharp edges, and the load forces acting on them, the diamond grains penetrate more or less deeply into the metal surface. Since the metal surface and the diamond grains move counter to each other, long scratches are produced on the metal surface. The abrasive mechanism is somewhat similar to the action of a turning tool. In the grinding procedures, abrasion is effected by diamond or silicon carbide grains quite firmly fixed in a bonding material, e.g. a resin, as is the case in the MD-Piano and MD-Primo disks. As soon as an abrasive grain is worn, it will be torn out of its resin bond and carried away together with the metal debris through the free spaces between the grinding spots. The grinding steps differ by grain size and therefore produce different scratch widths and scratch depths. The scratch depths are assumed to be less than one fifth of the scratch widths. In the fine grinding disks MD-Allegro and MD-Largo the diamond grains must be added and become partly fixed in the embedding material of the grinding hexagons and partly are free to roll. An abrasion process that uses rolling grains is called ‘lapping’. Thus the effective mechanisms of such disks are partial grinding and lapping. However, as those diamond particles which will not instantly become fixed in one of the resin hexagons are quickly carried away through the intermediate spaces, the process is considered to be predominantly grinding rather than lapping. The different scratch widths and depths are not only due to different grains sizes but also to the different hardness of the bonding media of the grinding disks. The polishing cloths are composed of fabrics of various fibre materials and textures. The dense texture of a pre-polishing cloth produces a low elasticity (resilience). Such a cloth is called a “hard” cloth. The diamond grains stay mainly on the surface and are not easily pushed into the interior of the cloth (cp. Fig. 11). “Soft” polishing cloths as used in the final polishing steps are smooth-faced or have a “nap” with flexible fibres that give the cloth a considerably increased resilience (see Fig. 12).

In polishing, the nap fibres with the diamond grains adhering to them give way under the specimen load and the manual forces exerted on the specimen and thereby produce very fine scratches only. On the other hand, it is just the elasticity of the diamond grain covered fibres which may produce a preferential abrasion of soft particles within a relatively hard matrix, i.e. lead to relief formation. Therefore, the duration of final polishing on very soft cloths is usually kept short. Of course, the total abrasion depth in a polishing step is considerably lower than in a grinding step and must not be intentionally increased by excessive pressure. Generally, the abrasive action on a polishing cloth is considered to be effected by fixed grains rather than by rolling grains because of the strong adhesion forces between the fibres and the small diamond grains and due to the partial penetration of the grains into the web. In all grinding and polishing processes the lubricant plays an important role, both by lubricating film formation and by its cooling effect. In the coarse grinding stages, water is efficient enough, whereas in the fine grinding and coarse polishing steps the use of a water based lubricant is expedient in most cases. In final polishing, however, a lubricant containing an oil component should be applied. In the processing of very soft metals, the use of a water based lubricant would favour deep deformation and pull-outs of hard inclusions from the material surface. Therefore, for such metals, an oil
based lubricant (e.g. the RED lubricant) should be used in all polishing stages. Nevertheless, even with very careful and sample-matched treatment, a certain residual deformation will remain after final polishing, which can be removed only by subsequent etching.

Conclusion
The underlying physical principles in grinding and polishing processes consist in a complex interaction between a material surface and the grinding and polishing media. The ideal result of metallographic sample preparation is a surface free of scratches, pullouts, relief formation, impressed grains and other artefacts that is suitable for microscopic examination. In most cases, however, except for some inclusions that are distinguished by their considerably different colour and/or reflectivity from the surrounding matrix, the true microstructure will be revealed only by a suitable etching.