The True **Microstructure** of **Materials**

Materialographic Preparation from Sorby to the Present
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Abstract
The microstructure expresses a high number of properties of a material. For this reason materialographic - or if we use the older, more common terminology, metallographic - preparation is important for both production - and research work.

In 1863 H. C. Sorby prepared the first "true structure" of steel, but for many years the importance of the preparation process was neglected, resulting in structures with many artifacts. In the 1930s the American J.R. Vilella takes up the preparation of a "true structure" and in the 1950s to 70s the Australian L.E. Samuels describes in a conclusive way, how to obtain a true structure by mechanical methods.

In mechanical preparation, the removal rate and deformation are decisive factors for obtaining a true structure or one with a minimum of artifacts.

Prior to World War 2 hand preparation with Al_2O_3 (emery paper and alumina) was used. In the 1950s semi-automatic machines with rotating discs were developed, using SiC and diamond as grinding/polishing media.

Preparation artifacts in the structure might create an "untrue" structure which can mistakenly be accepted as "true". A number of examples are given.

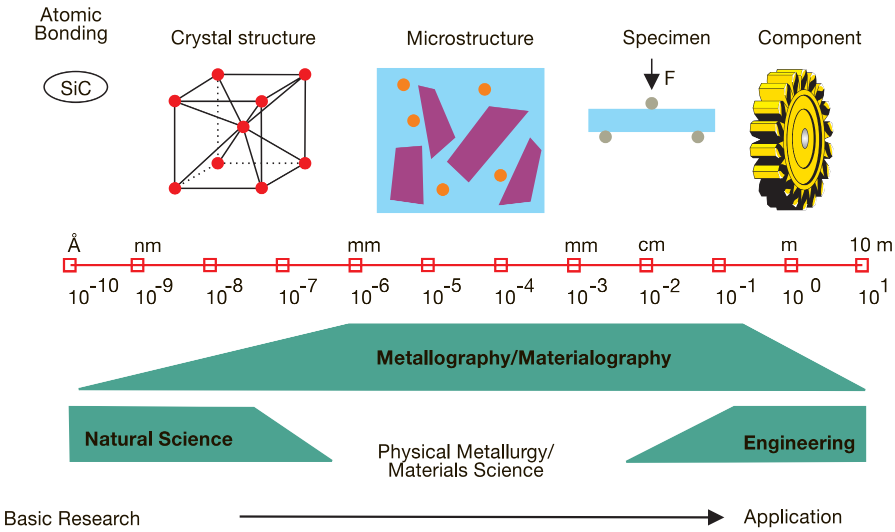

Fig. 1

Materialography/Metallography
For the title of this article I have used the word MATERIALOGRAPHY, instead of the usual METALLOGRAPHY. Considering that we work with not only metals, but ceramics, plastics, electronic parts etc, MATERIALOGRAPHY could be a more correct name. As early as 1968 Crowther and Spanholtz wrote an article in Metal Progress called "A new name for Metallography? Try Materialography" (1).

Materialography covers a very wide field in material investigation. Materialography bridges the gap between science and engineering (Fig.1) (2).

The microstructural parameters strongly influence many of the properties of a material (Fig.2)(2). Each material contains many millions of microstructural features per cubic centimetre. These microstructural features can exist in sizes spanning more than ten orders of magnitude (Fig. 1). Today there is a whole range of instruments with which nearly all of the features across this range can be made visible. In this sense the expression **Continuous Metallography** or rather **Continuous Materialography**, has been coined (3), covering examination from the macro to the atomic level.

The microstructure we see in the microscope is two dimensional, but we should not lose sight of the fact that the constituents in a material are three dimensionally arranged.

A photo montage shows the prepared surface with the microstructure and the material below the surface (Fig.3)(2). It can be seen that the true size of the grains cannot be deduced from the microstructure. A statistic extrapolation of a microstructure with elongated grains shows that based on the two dimensional surface, approx. 80% of the grains are relatively short and have an almost equiaxial shape. An extrapolation of the grain population in three dimensions, however, reveals a much higher variation in grain length and the average grain length is longer.

Having realized the limitations of the two-dimensional structure, we can establish the fact that the examination of the microstructure is of enormous importance. From now on I shall concentrate on the quality of this structure.

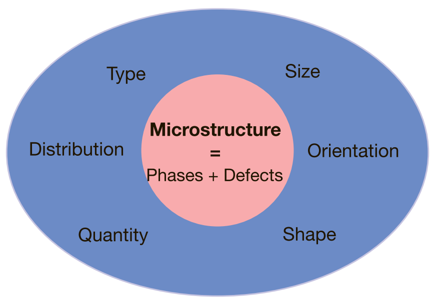

Fig. 2

The True Structure

Historical. Before we go into a more exact definition of the true structure, we shall look at one of the first true structures pro-duced. In 1863-65 H.C. Sorby made a number of preparations of steel; an example is a structure of Bessemer steel containing ferrite and pearlite (Fig.4)(4). We know today that this structure is correct, a true structure without artifacts. H.C. Sorby was fully aware that to obtain a true structure, the surface must be treated very carefully.

After Sorby it seems that nobody really paid attention to the preparation of the true structure for many years.

The American J.R. Vilella in the 1930s seems to be the first to establish a definition of the true structure and point out the importance of a correct preparation, avoiding surfaces with distorted metal (6).

He showed how a structure at that time, known as “sorbite” or “troostito - sorbite” is normal pearlite severely distorted during polishing (Figs.5 and 6).

Based on the work by Vilella, the Australian L.E. Samuels since the 1950s established the conclusive work on metallographic preparation by mechanical methods. He pointed out the importance of the preparation process to obtain a true structure. Without this the best examinations and inspired interpretations of the structure will be of no avail (5).

Definition of the True Structure. Based on Vilella and Samuels the true structure can be defined: (7)

No deformation
No scratches
No pull-outs
No false structures (artifacts)
No introduction of foreign elements
No smearing
No relief or rounded edges
No thermal damage

The preparation process will always influence the prepared surface. This influence is concentrated in certain parts of the surface, “danger zones”, where the risk of artifacts is higher (Figs. 8 and 9).

With mechanical polishing an approximate true structure can be obtained when the correct procedures are followed, even with very heterogeneous materials, whereas electrolysis may create problems if more than one phase is present in the structure during electrolytic polishing.

Considering that most materials are heterogeneous (or non-conductive), mechanical polishing is by far the most used method, and this will be the only one discussed in the following text.

Mechanical Preparation

Two features should be considered during mechanical preparation:

Removal rate. The rate at which material is removed from the surface of the specimen.

Deformation. The nature and depth of the plastically deformed layer that is produced in the specimen surface.

In general we want the removal rate to be as high as possible and the deformation to be as low as possible. This depends on the interaction between the abrasive grain and the specimen surface.

Removal Rate - Grinding. Grinding is the first stage in the preparation process. When preparing a specimen both cutting and grinding involve abrasives, either fixed in a bond as on a cut-off wheel or fixed to a coated surface like grinding pa-per. In the case of composite discs, the abrasive grains are added to the surface in a free flow, but we shall see later that even in this case, the grain - when cutting action takes place - is fixed. Silicon carbide (SiC), aluminium oxide (Al_2O_3) and diamond are mainly used as abrasives.

Influence of Grinding Process on Removal Rate. Three modes of interaction can be recognized (5):

Lapping**:** The grain rolls between the specimen and the preparation disc (Fig.9).

A corner of the grain digs into the specimen and turns, leaving a small cavity with strong deformation. The removal rate is

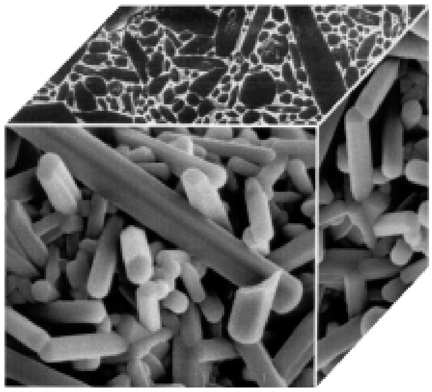

Fig. 3. Photo-montage of a micro section of silicon nitride alloy superimposed upon a pile of silicon nitride crystallite

Fig. 4. Original Sorby specimen 1863, Bessemer steel 0.2% carbon. BF, 450:1

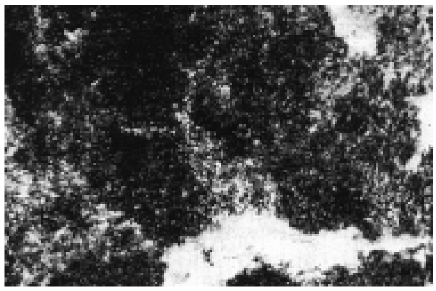

Fig. 5. “troostito-sorbite”. BF, 1000:1

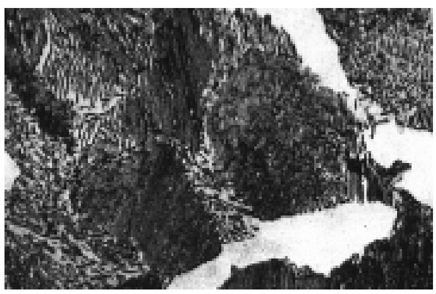

Fig. 6. Same as Fig. 5, normal pearlite. BF, 1000:1

very low and lapping is not suited for metallographic preparation.

Grinding: The grain is fixed and acts as a machine tool, because the rake angle is correct for cutting a chip (Fig.10). The rake angle can be positive, 0 or negative, and grinding only takes place when the angle is positive, 0 or to a certain degree negative. At a given negative angle, the critical rake angle, the chip is not made anymore and the grain starts "plowing" instead of cutting. When grinding, the efficiency of material removal approaches 100%.
Plowing: When plowing, the rake angle is so negative that only a groove is made in the specimen surface (Fig. 10). A standing wave bulge forms in front of the grain, and material is displaced into a ridge, on each side of the groove. The removal rate approaches zero.

As well as the rake angle, the geometric shape of the grain is also important (5). The most effective shape is a V-form, creating an efficient chip, provided that the rake angle is correct. If the grain is flat, the cross-section of the chip is reduced and in the case of flat grains of a certain size, the specific pressure between grain and surface will decrease and no cutting will take place, resulting in plowing or no action at all.

Grinding surfaces. We have so far identified two important parameters, rake angle and grain shape.

We can now relate these to the two grinding surfaces, waterproof SiC paper, which is commonly used and a surface with diamond grains.

At 220 grit SiC paper we see the different angles and shapes and it is evident that also the grain size varies widely (Figs.11 and 12)(5)(8). At 220 grit, the average grain size is 59 µm and the largest allowed grain size is 74 µm. Because of this large variation in rake angles, shape and size, only 1 or 2% of the many visible grains of the grinding paper, actually remove material by cutting a chip. About the same proportion produce scratches in the surface by plowing. The remainder have no effect (5).

During the process the SiC grains are broken down and after a period of time most grains will be relatively flat from fracturing and wear so that cutting a chip will change into plowing.

Using a diamond grinding disc as the grinding surface, with the diamonds partly embedded in a bond, the process is somewhat different from the grinding paper (Fig.13). The fracturing is lower, partly because the diamond grains are not as brittle as the SiC and partly because the grain is fixed in the bond. It appears that the diamond grains composed with facets, constitute effective cutting points (5), which give a higher removal rate. The diamond grinding disc normally has a constant removal rate over a long period of time, as long as the surface is not clogged by ground-off material. This is avoided with an efficient flow of grinding fluid and a configuration of the surface so that the debris is channelled away from the grinding surface.

Composite discs (platens) are mostly used for the fine steps in grinding. The surface consists of a resin reinforced with a metal powder, and the abrasive, mostly diamond is added during the process.

It is very important that the surface is able to fix the abrasive grain, establishing a grinding process (Fig.14). If a large portion of the grains are rolling between the disc surface and the specimen surface, a lapping action is established causing a very low removal rate and heavy deformation.

Heating of the specimen surface might cause artifacts and if debris is not removed, the clogged grinding surface will cause severe damage to the specimen surface.

Removal Rate - Polishing. Polishing is the last stage in mechanical preparation. It is very important for two reasons, the deformation from grinding must be removed (rough polishing), and the surface established after the last polishing step (final polishing), should preferably represent the "true structure". Alumina (Al_2O_3) in different grain sizes and crystal structures were - for many years - the most used abrasive for polishing.

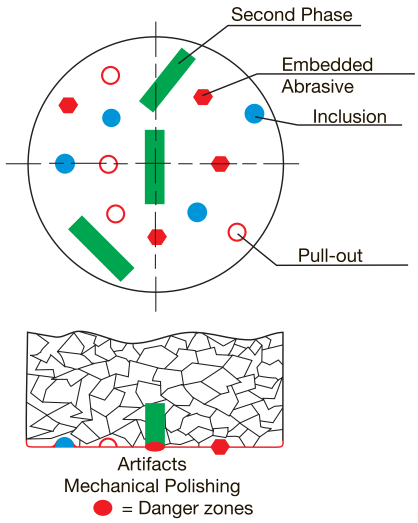

Fig. 7. Mechanical polishing. "Danger zones"

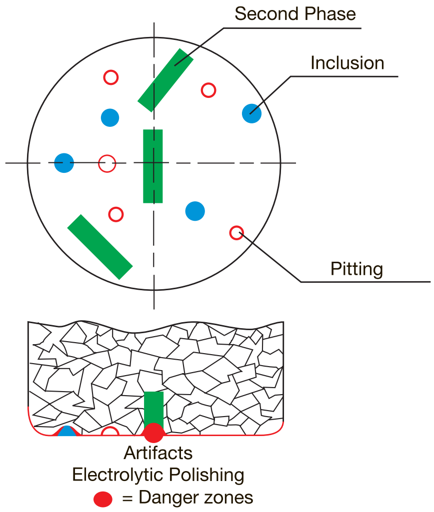

Fig. 8. Electrolytic polishing. "Danger zones"

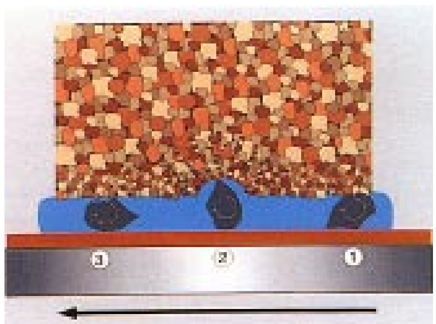

Fig. 9. Lapping

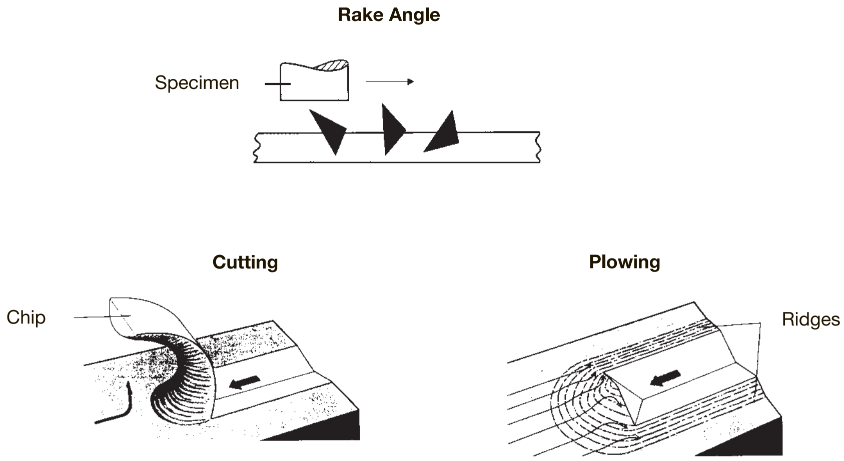

Fig. 10. Rake angles, cutting, plowing

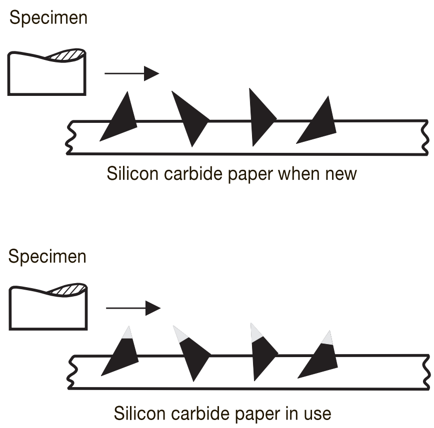

Fig. 11. Grinding on SiC paper

Using very fine grain sizes, 0 - 1µm and 0 - 0,1µm after grinding, results in a low removal rate, making it impossible to remove the deformed layer created by grinding. In the 1930s, Vilella (6) pointed out the importance of rough polishing, using 600 grit alumina on a cloth impregnated with paraffin.

With the introduction of diamond as an abrasive, the removal rates were increased considerably (5), whilst at the same time introducing less deformation and a better overall quality of the specimen surface (10).

Polishing takes place on polishing cloths, mostly textiles, ranging from very hard and plane to very resilient with a nap.

Influence of Polishing Process on Removal Rate. The polishing process and the grinding process are in principle the same (5).

This means that at polishing an abrasive grain is able to produce a chip, implying that the grain is, at least momentarily, fixed in the polishing cloth (Fig.15).

The diamond grain wedges between the fibres with a rake angle sufficient to be able to cut a chip from the specimen surface.

To obtain a high removal rate at rough polishing, hard cloths are used, creating a higher load on the grain, giving a larger chip.

Using softer, more resilient cloths for the final steps, ensures smaller scratches and less deformation of the surface.

Influence of Polishing Abrasive on Removal Rate. As for grinding the hardness of the abrasive is important. Therefore diamond is used for both rough polishing and polishing. For final polishing alumina (Al_2O_3), colloidal silica (SiO_2) and Magnesia (MgO) are also used.

Likewise, the shape of the grain plays a role. Polycrystalline diamonds give a higher removal rate than monocrystalline diamonds (5), probably because the individual polycrystalline grain contains more angular points of the size needed to provide cutting points than the monocrystalline ones.

Influence of Polishing Fluid on Removal Rate. The fluid added during the polishing process has an important influence on the removal rate. In most cases fluids based on a hydrocarbon like kerosine (5) give the

Fig. 12. SiC paper 220 Grit (FEPA), SEM

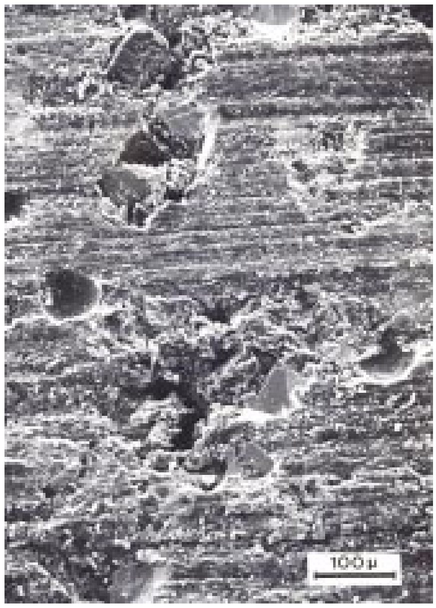

Fig. 13. Diamond grinding disc "220 Grit", SEM

highest removal rate. A similar effect can be obtained using an alcohol based fluid. Fluids are also important for surface cooling.

Deformation - Grinding. The separation of a chip during machining operations induces complex systems of plastic deformation in both the separating chip and the specimen material. An inevitable consequence is that a layer plastically deformed during machining, is left in the new surface that is produced. In general terms, the strains in this layer are very large at the surface and decrease more or less exponentially with depth.

This deformed layer becomes important in metallography, when the plastic deformation changes the microstructure of the specimen in a way that can be detected in the particular microscopic examination that is to be carried out. The layer is then an important potential source of false structures, or *preparation artifacts*, the avoidance of which is one of the primary objectives of a materialograhic preparation sequence (5).

Influence of Grinding Process on Deformation. During grinding the abrasive grains act as machine tools set at different rake angles. When a chip is separated from the surface, the shear strains are concentrated in the so-called shear zone in front of the tool. A region adjacent to this shear zone, and extending into the specimen in advance of the tool is also plastically deformed, though to a lesser degree (Fig. 16).

Samuels (5) has done an exhaustive study of the deformation created in the specimen

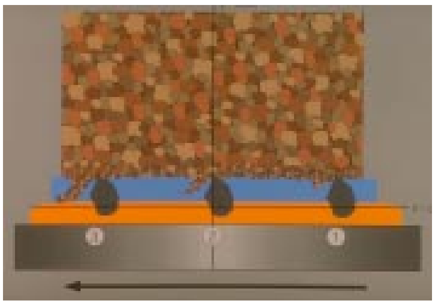

Fig. 14. Grinding on a composite disc

surface, by using taper sections. A taper section is produced by preparing the specimen at a small angle, the taper angle. When using a taper angle of 5°44' a 10 x enlargement it obtained, making it possible to analyse a greater surface structure detail under an optical microscope (9).

A taper section of 70:30 brass ground on a 220 grit SiC paper, shows the surface of the specimen with scratches and the deformation below (Fig.17).

Samuels (5) split up the layers into three levels of deformation:

Depth of fragmented layer (Df)
Approximately equal to the depth of the surface scratches.

Depth of deformation (Dd)
Maximum depth beneath the root of the surface scratches to the elastic-plastic boundary (Fig 18 (d)).

Depth of significant deformation (Ds)
Maximum depth beneath the root of the surface scratches of the deformation that will noticeably affect the observations to be made on the finished surface.

Example: Annealed polycrystalline 70:30 Brass, SiC grinding paper, 220 grit, with water

Df (scratches):	2 µm
Dd:	77 µm
Ds:	7.5 µm

The fragmented layer consists of severe plastic deformation and is easily recognized. In the deformed layer, the material can be modified in different ways, such as strain induced transformation (austenitic steel) or massive twinning (polycrystalline zinc).

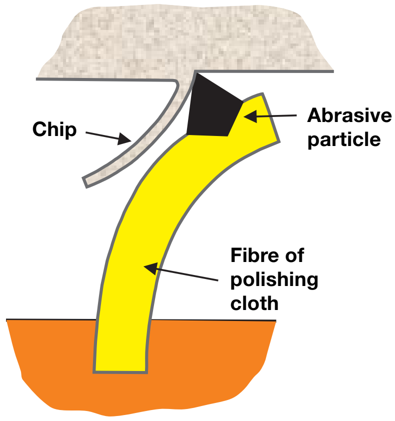

Fig. 15. Abrasive grain fixed in polishing cloth

Fig. 16. Section of a chip cut in 70:30 brass by an orthogonal tool with a highly negative rake angle (5)

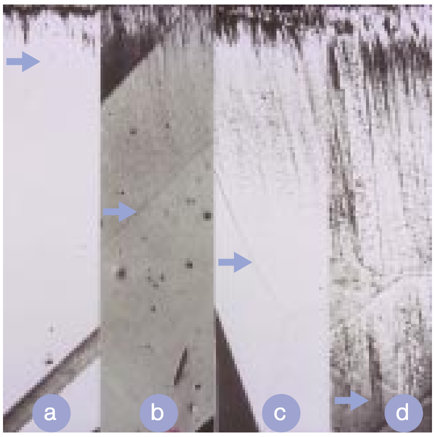

Fig. 17. Taper section of the surface of annealed polycrystalline 70:30 brass that has been ground on 220 grit SiC paper. The section has been etched by several methods that have different threshold strains for revealing deformation as follows. (a) Ferric chloride reagent (threshold strain: 5% compression). (b) Cupric ammonium chloride reagent (threshold strain: 0.1% compression). (c) Low sensitivity thiosulfate etch (threshold strain: 0.1% compression. (d) High-sensitivity thiosulfate etch (threshold strain: elastic limit). In each case, the base of the layer in which the manifestations of deformation have been developed is indicated by an arrow. Taper ratio, 8.2. 250:1 (5)

Deformation - Polishing. Polishing, in principle being the same process as grinding, also produces deformed layers, only shallower.

Influence of the Polishing Process on Deformation. The depth of the deformed layers is an order of magnitude smaller than that on surfaces ground with SiC paper (Fig.18)(5).

In principle even the finest abrasive will create a deformed layer. With very fine abrasives like alumina, Al_2O_3 and silica, SiO_2, for the final polishing step, a very clean surface can be obtained. Silica has a grain size of a fraction of a micron creating a combined mechanical and chemical material removal.

In the literature, the very thin deformed layer left by the last polishing step is often called the Beilby Layer (5)(11).

Preparation Process. The preparation process can be defined as the process including grinding and polishing.

We see schematically how the scratches and the deformation layer are induced from step to step (Fig. 19). It is important that both scratches and the significant deformation are removed in each subsequent step. It can be seen that rough polishing is a vital step, as the relatively large deformation layer from the last step in grinding, has to be removed.

Grinding/Polishing Machines. Sorby worked by hand without the benefit of rotating surfaces, it took approx. five weeks to prepare a steel specimen.

The Englishman, I. E. Stead (1851-1923), developed equipment far less time-consuming than Sorby's (12), but the rotating wheel was probably not used until the beginning of this century. Preparation by hand using emery paper for grinding and alumina for polishing was common until after World War 2. With the increase in the number of metallographic specimens, the need for better machines was evident, and semi automatic and automatic machines both for mechanical and electrolytical preparation were developed.

With the machines, hand preparation was avoided and the reproducibility, necessary for quality control, was greatly increased. Today most specimens are prepared mechanically in a specimen holder with either fixed or loose specimens.

Preparation Artifacts
An artifact is a false structure introduced during the preparation of a surface (5).

A number of artifacts caused by the process have already been mentioned; scratches, deformation, smearing, and twins. The list is considerably longer: Micro cracks, pull-outs, edge rounding, relief, comet tails, contamination, embedded abrasives and lapping tracks, all artifacts caused by the preparation process. Artifacts can also be introduced during chemical etching of the surface. Most of these artifacts can be readily observed under the microscope. The metallographer can decide whether, for example, a scratch is accepted as it does not disturb the structural analysis, or whether the specimen surface should be re-prepared.

In some cases it is difficult to establish the truth, e.g. pores in the structure, could be pull-outs or "true". A number of such "true" and "untrue" structures will be discussed below.

Abrasive	Grade µm/Grit	Df µm	Ds µm	Dd µm
SiC paper	220	2.0	7.5	77
SiC paper	400	1.5	6.5	43
SiC paper	600	0.8	5.0	22
Diamond	6	0.17		1.0
Diamond	1	0.1		0.7
Alumina, α-type	0-1	...		2.5
Alumina, γ-type	0-0.1	0.03		0.7

Fig. 18. Annealed polycrystalline. 70:30 brass. Depth of plastically deformed layer (5)

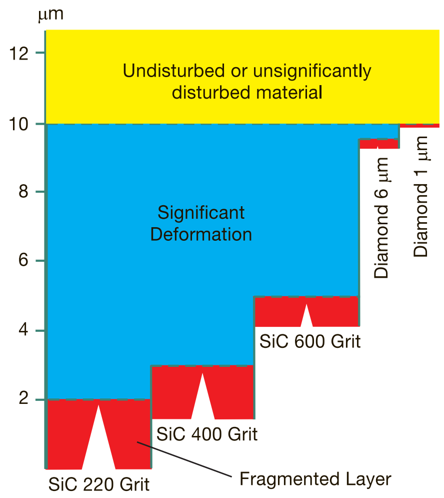

Fig. 19. Preparation process. Annealed polycrystalline70:30 Brass (5)

Reproducibility
Reproducibility is of utmost importance both in research and production.

Using e.g. image analysis is of no use, if the preparation process is not able to produce reproducible results. An example of quality control of a material is pores in a plasma spray coating. Relatively small variations in the process or in the consumables will cause a variation in the number of pores to be seen under the microscope.

"True" and "Untrue" Structures
In the following an "untrue" structure is defined as a structure, which in principle could be correct; only the experienced metallographer is able to see that the structure might have one or several artifacts.

Plasma spray coatings. Materialographic examination is very important to establish the quality of a plasma spray coating. A number of parameters can be examined: porosity, cracks, amount of oxide, interface contamination, unmelted particles and microhardness.

With plasma coatings it is very important to obtain a true structure, as the coatings are often used for high technological applications, like turbine blades for jet engines. This means that the quality requirements must be met whilst at the same time avoiding waste of valuable components.

With plasma spray coatings we often see two artifacts, smearing and pull-outs.

Material: 88/12 WC/Co (13). This coating is very hard due to the WC particles, but the Co matrix is relatively ductile. It means that when using finer SiC grinding papers, the WC particles are moved into the pores creating a "dense" structure (Fig.20).

This was considered the correct structure for quite a long time, because a "dense" structure is preferred to a porous structure. Only through a controlled process using different examination methods it could be decided that the structure with a much higher porosity (approx. 11%) was the true structure (Fig.21). By using SiC paper for the plane grinding only, and a composite disc with diamonds for fine grinding, the smeared layer was removed because of the superior hardness of the diamond abrasive. Following steps with 6 µm and 3 µm on hard cloths also secured that smearing and pull-outs were avoided.

Material: ZrO_2 (14). The artifact experienced with this material is pull-outs because it is very hard and brittle. During the first preparation steps a lot of pull-outs take place and the porosity level is very high (Fig.22).

If the preparation is stopped too early (Fig. 23), the level of porosity is too high, although the polishing times would be considered "normal". To remove the pull-outs being created at each step, the specimen must be treated until the number of pull-outs remains constant in each step. This gives considerably longer polishing times (Fig.24).

Ceramics. Pull-outs are a very serious artifact with ceramics, which are hard and brittle.

Material: Al_2O_3 (15). Many pull-outs will occur during plane grinding and it is very important that the fine grinding step has a high removal rate so that the damaged layer is removed. If the composite disc used for fine grinding is not working properly, in this case not plane, the damaged layer is not removed and even when the following steps are correct, the result is a

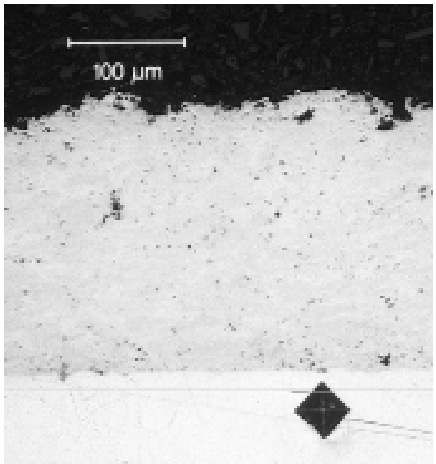

Fig. 20. Plasma spray coating 88/12 WC/Co, "dense" structure, incorrect preparation.

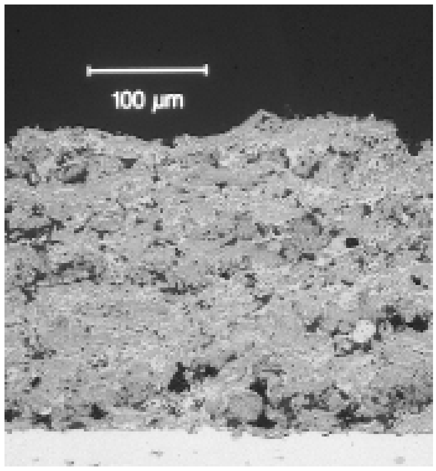

Fig. 21. Same as Fig. 27, correct preparation.

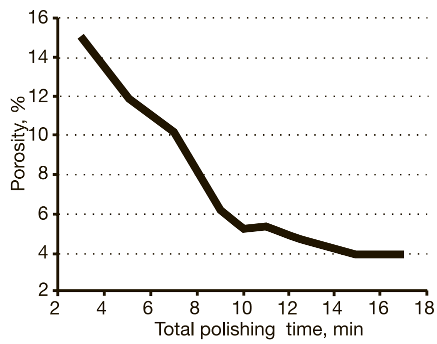

Fig. 22. ZrO_2, porosity level during polishing

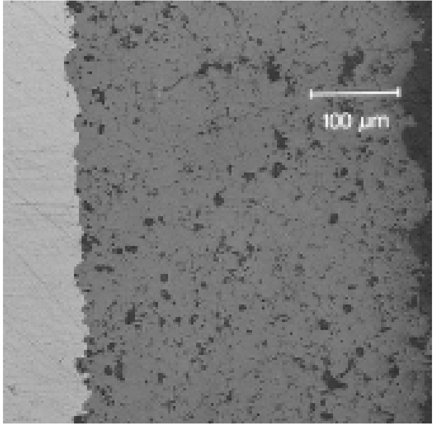

Fig. 23. ZrO_2, porosity too high after polishing in 7 min.

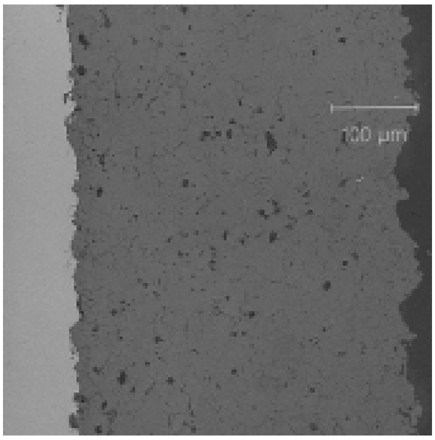

Fig. 24. Same as Fig. 30, correct porosity after polishing in 15 min.

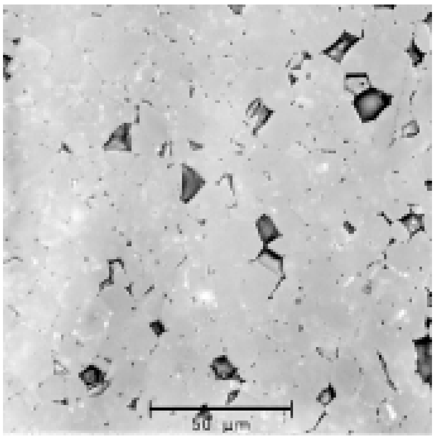

Fig. 25. Al_2O_3, many pull-outs, incorrect preparation.

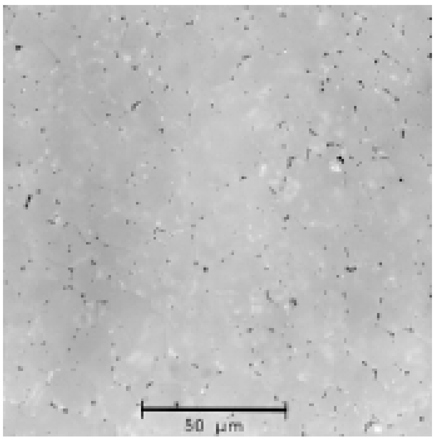

Fig. 26. Same as Fig. 32, correct porosity, correct preparation

structure with many pull-outs, an apparent "high porosity" (Fig.25).

The same specimen prepared in the same way with a plane, efficient, composite disc, has the correct porosity (Fig.26).

Grinding artifacts. These artifacts are the "classic" example of deformation, mostly seen in relatively soft materials. Scratch traces, traces of plowing, lapping tracks and a distorted surface, as described by Vilella (6) (Fig. 5), are present, but not recognized, when the preparation is finished. Only an etching of the surface reveals the artifacts.

The main reason for these artifacts is a too short (or missing) rough polishing after the last grinding step with SiC paper (Fig. 19). Modern preparation uses composite discs or special „cloths" for fine grinding, and not SiC paper. Fine grinding is therefore performed without inducing the heavy defor-mation as SiC paper, making it possible to shorten or totally avoid the rough polishing step.

Material: Low carbon steel with inclusions. The surface "as-polished" seems acceptable, showing the inclusions reasonably well with a few scratches.

When etched, many scratch traces and traces of plowing are visible and the ferrite grains are distorted as shown on the "sorbite" structure (Fig. 5).

After preparation with correct fine grinding and rough polishing, the etched surface is totally free from all artifacts mentioned.

The Metallographer's Rule of Thumb
As a conclusion we can establish a Rule of Thumb for obtaining the True Structure: PUT UP A GOAL FOR EACH PREPARATION STEP AND ACHIEVE IT (16).

When establishing a new preparation method a goal should be established for each step, do not move on to the next step until this goal has been achieved.
In the rough/plane grinding steps it may be to make the surface flat or to remove a certain amount of material.

The goal during a fine grinding step may be to remove the rough grinding damage and to make the surface super flat. This step and the next will be decisive for obtaining a true structure.

The rough polishing goal may be to remove damage to inclusions or to brittle layers, keep the flatness and remove grinding deformations or embedded abrasive grains.

Polishing may again aim at removing damage from certain areas of the surface, removing smearing and "brightening-up" the surface and providing contrast.

The goal in final polishing may be to remove all mechanical deformation, to polish up the soft spots (e.g. graphite) or to take the structure into relief for "optical etching".

Example: Composite with SiC fibres in a Ti-alloy matrix (Fig.27).

Fine grinding: Goal: Establish the ceramic fibres and obtain a flat surface. The matrix will be taken care of at the following steps.

Rough polishing: Goal: Remove damage to the matrix, establish a flat surface with a uniform scratch pattern.

Polishing: Goal: Keep the established surface, but with a finer scratch pattern.

Final Polishing: Goal: Remove (most of) of the scratches without creating relief between the ceramic fibres and matrix.

Materialography - The Future
In production (quality control), the need for reliable, reproducible preparation will probably increase as a consequence of quality standards, ISO 9000 and QS 9000. This leads to more automatic equipment and more efficient and uniform consumables.

In research, "the true microstructure" is important for the development of new materials, expressed by G. Petzow (17): "With less resources, less energy and a smaller environmental impact, we must attempt to make today's highest standards of living accessible to all people, based on more intelligent, innovative materials and

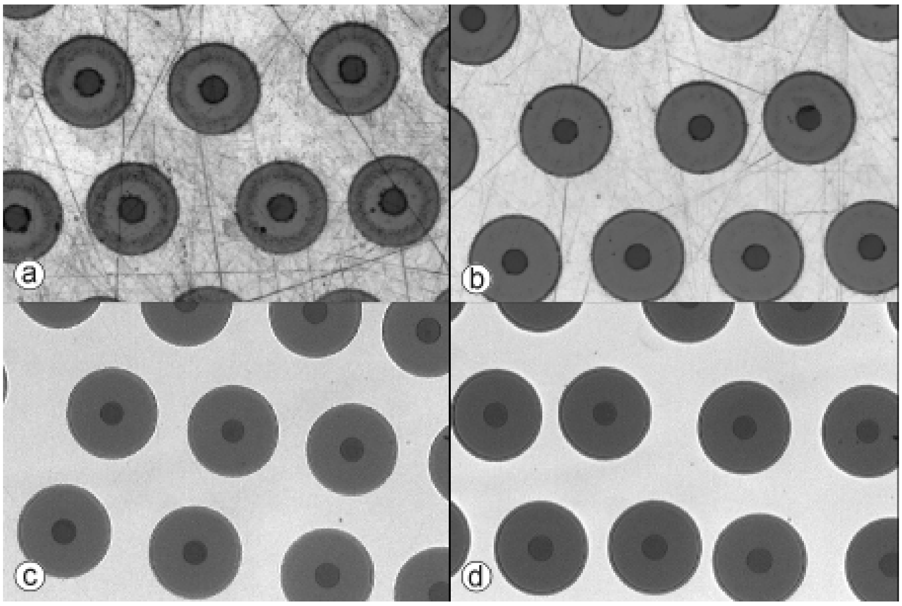

Fig. 27. SiC Fibres in Ti-alloy matrix, (a) after fine grinding, (b) after rough polishing, (c) after polishing, (d) after final polishing. BF, 100:1

technologies". - "All of these materials have to be synthesized, produced and optimized by *mastering the microstructure* and phase relations to get the best properties for a specific application*".*

Summary

The goal for all materialographic preparation must be the true structure. The true structure has been defined and the removal rate and deformation at mechanical preparation described and discussed.

At the mechanical preparation the abrasive grains act as machine tools, removing material and at the same time creating artifacts. By using the correct abrasives, polishing cloths and - fluids, a true structure can be obtained.

A number of artifacts can be developed during the process creating "untrue" structures, which can be interpreted as "true". A number of "untrue" and "true" structures have been described showing the importance of the metallographer's correct interpretation.

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